UDC 661.862 : 620.1

Ref. No. : ISO/R 803-1968 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 803

ALUMINIUM OXIDE PRIMARILY USED FOR THE PRODUCTION OF ALUMINIUM

DETERMINATION OF LOSS OF MASS AT 300 °C

(CONVENTIONAL MOISTURE)

1st EDITION
August 1968

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

Printed in Switzerland

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

BRIEF HISTORY

The ISO Recommendation R 803, *Aluminium oxide primarily used for the production of aluminium – Determination of loss of mass at 300 °C (Conventional moisture)*, 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 by the Technical Committee began in 1961 and led, in 1964, to the adoption of a Draft ISO Recommendation.

In July 1966, this Draft ISO Recommendation (No. 1025) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria	India	South Africa, Rep. of
Belgium	Ireland	Spain
Brazil	Israel	Sweden
Bulgaria	Italy	Switzerland
Canada	Japan	Turkey
Chile	Korea, Rep. of	U.A.R.
Czechoslovakia	Netherlands	United Kingdom
France	Norway	U.S.A.
Germany	Poland	U.S.S.R.
Hungary	Romania	Yugoslavia

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in August 1968, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 803 August 1968

ALUMINIUM OXIDE PRIMARILY USED FOR THE PRODUCTION OF ALUMINIUM

DETERMINATION OF LOSS OF MASS AT 300 °C

(CONVENTIONAL MOISTURE)

INTRODUCTION

Depending on its degree of calcination, aluminium oxide shows a tendency to readsorb variable quantities of water by a process involving physical forces (residual activity).

The water readsorbed cannot be completely eliminated by simply drying at 105 °C, the temperature usually employed for the determination of moisture. Therefore, it is necessary to use a suitably higher drying temperature. It is accepted that elimination of water is almost complete at 300 °C and this temperature is conventionally adopted.

1. SCOPE

This ISO Recommendation describes the method for the determination of loss of mass on drying at 300 °C (conventional moisture).

2. PRINCIPLE

Drying at 300 °C for 2 hours and determination of loss of mass.

3. APPARATUS

Ordinary laboratory apparatus and

3.1 *Desiccator*, preferably containing freshly activated alumina or phosphorus pentoxide (the use of calcium chloride should be avoided).

3.2 *Weighing bottle*, squat form, approximately 45 mm in diameter.

3.3 *Electric oven*, ventilated by convection and controlled at 300 ± 10 °C.

4. PROCEDURE

4.1 Test portion

Weigh to the nearest 0.001 g, approximately 5 g of the crude sample* in the weighing bottle (3.2), previously dried at 300 °C and tared.

* See ISO Recommendation R 802, *Aluminium oxide primarly used for the production of aluminium – Preparation and storage of test samples*, clause 2.2.

4.2 Determination

Place the uncovered weighing bottle and its lid in the oven (3.3) and heat for 2 hours. Remove weighing bottle and lid from the oven and place in the desiccator (3.1) without covering the weighing bottle completely. After 30 minutes cooling, cover the weighing bottle and weigh it.

5. EXPRESSION OF RESULTS

The loss of mass at 300 °C is expressed as a percentage (m/m) by the following formula :

$$\frac{m_2 - m_1}{E} \times 100$$

where

m_2 is the mass, in grammes, of the weighing bottle containing the test portion before drying,

m_1 is the mass, in grammes, of the weighing bottle containing the test portion after drying,

E is the mass, in grammes, of the test portion.

6. TEST REPORT

Give the following particulars :

(*a*) the reference to the method used,

(*b*) the results and the method of expression used,

(*c*) any unusual features noted during the determination,

(*d*) any operation not included in this ISO Recommendation or regarded as optional.

Date of the first printing :
November 1968

Price : Sw. fr. 4.50

ISO Recommendation R 804 August 1968

ALUMINIUM OXIDE PRIMARILY USED FOR THE PRODUCTION OF ALUMINIUM

PREPARATION OF SAMPLE SOLUTION FOR ANALYSIS

1. SCOPE

This ISO Recommendation describes the method of preparation of a sample solution in order to obtain a principal solution (solution P) for certain chemical tests.

2. PRINCIPLE

Alkaline fusion of aluminium oxide

- 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 (see clause 6.1) so that the pH of the final solution is approximately 1, after dilution to 500 ml, or approximately 0.4, after dilution to 250 ml.

3. REAGENTS

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

3.1 *Sodium carbonate,* anhydrous.

3.2 *Boric acid* (H_3BO_3).

3.3 *Sodium tetraborate,* anhydrous ($Na_2B_4O_7$).

3.4 *Aluminium oxide,* pure.

3.5 *Nitric acid,* approximately 8 N solution. Dilute 540 ml of approximately $d = 1.4$ nitric acid solution (68 % m/m solution) with water and dilute to 1000 ml.

4. APPARATUS

Ordinary laboratory apparatus and

4.1 *Platinum flat-bottomed dish,* approximately 70 mm in diameter and 35 mm deep, fitted with platinum lid.

4.2 *Electric furnace,* controlled at 500 ± 50 °C.

4.3 *Electric furnace,* controlled between 1000 and 1025 °C.

5. PROCEDURE

5.1 Test portion

Weigh to the nearest 0.001 g, exactly 5 g of sample dried at 300 °C *.

5.2 Fusion of test portion

Weigh into the platinum dish (4.1)

- either 12 g of sodium carbonate (3.1) and 4 g of boric acid (3.2),
- or 10.3 g of sodium carbonate (3.1) and 3.3 g of sodium tetraborate (3.3).

Thoroughly mix. Add the test portion (5.1) and mix carefully the whole, preferably with a platinum spatula. Cover the dish with its lid and place it in the electric furnace (4.2) controlled at 500 ± 50 °C, taking care to isolate it from the bottom 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 (4.3) controlled between 1000 and 1025 °C, taking care to isolate it, as before, from the bottom of the furnace.

Keep the dish in the furnace for 30 minutes. The operator should make sure that a temperature of between 1000 and 1025 °C is kept for a minimum of 20 minutes.

5.3 Preparation of the principal solution

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

After slight cooling, transfer the contents of the dish to a polyethylene beaker of suitable capacity containing 50 ml of the nitric acid solution (3.5) (see Note 1).

Dissolve the residue still attached to the dish walls (consisting essentially of ferric oxide, calcium oxide and titanium oxide) with 20 ml of the nitric acid solution (3.5) 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 content 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 luke-warm, transfer to a 250 or 500 ml one-mark volumetric flask depending on the content of the elements to be determined. Cool and dilute to the mark (solution P) (see Note 2).

5.4 Blank test

Together with the analysis carry out a blank test using the same quantities of all reagents, and operating in the presence or in the absence of pure aluminium oxide (3.4) according to the determination to be carried out.

5.4.1 *Blank test in the presence of pure aluminium oxide.* Follow the same procedure as used for the test sample (clause 5.2 and 5.3) using exactly 5 g of pure aluminium oxide (3.4) weighed to the nearest 0.001 g.

---

* See ISO Recommendation R 802, *Aluminium oxide primarily used for the production of aluminium – Preparation and storage of test samples*, clause 2.3.