INTERNATIONAL STANDARD 2961

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

Aluminium oxide primarily used for the production of aluminium — Determination of an adsorption index

Oxyde d'aluminium principalement utilisé pour la production de l'aluminium — Détermination d'un indice d'adsorption

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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.

International Standard ISO 2961 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in October 1972.

It has been approved by the Member Bodies of the following countries :

Austria	India	Romania
Belgium	Israel	South Africa, Rep. of
Czechoslovakia	Italy	Sweden
Egypt, Arab Rep. of	Netherlands	Thailand
France	New Zealand	Turkey
Germany	Poland	U.S.S.R.
Hungary	Portugal	

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

The Member Body of the following country expressed disapproval of the document on technical grounds :

Switzerland

Printed in Switzerland

Aluminium oxide primarily used for the production of aluminium — Determination of an adsorption index

0 INTRODUCTION

Any industrial calcined aluminium oxide possesses, depending on its texture, a greater or lesser power of adsorption of certain reagents, vapours or gases.

The adsorption of a vapour or of a gas on a sample of aluminium oxide follows the general laws of adsorption on powdered solids.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies two methods for the determination of an adsorption index for industrial calcined aluminium oxide.

The first method, using carbon tetrachloride vapour, is applicable if the density is higher than 3,75 g/ml. The second, using water vapour, is applicable if the density is lower than 3,75 g/ml.

2 REFERENCE

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

3 PRINCIPLE

Evaluation of the adsorptive power by exposure of a previously dried sample of aluminium oxide to an atmosphere saturated with carbon tetrachloride vapour or water vapour at a conventional temperature and measurement of the quantity of carbon tetrachloride vapour or water vapour adsorbed after a conventional time.

4 FIRST METHOD, FOR ALUMINIUM OXIDE OF DENSITY HIGHER THAN 3,75 g/ml

4.1 Reagent

4.1.1 Carbon tetrachloride, ρ 1,594 g/ml, boiling point 76,7 °C.

4.2 Apparatus

Ordinary laboratory apparatus and :

4.2.1 Weighing bottle, squat form, of borosilicate glass, with ground glass cover, diameter 45 mm and height 28 mm (30 mm with cover).

4.2.2 Electric oven, with natural draught, capable of being controlled at 300 ± 10 °C.

4.2.3 Desiccator, preferably with freshly activated alumina or phosphorus pentoxide, but not calcium chloride.

4.2.4 Glass vessel, for use as a desiccator, the dimensions of which are shown in the figure, fitted with a stainless steel support plate, perforated by 10 circular apertures evenly distributed as shown in the figure, containing about 200 ml of the carbon tetrachloride (4.1.1), and placed inside an enclosure at 25 ± 0,5 °C.

4.3 Procedure

4.3.1 *Test portion*

Dry the weighing bottle (4.2.1), fitted with its cover, for 2 h in the electric oven (4.2.2) controlled at 300 ± 10 °C. Remove it from the oven, place it in the desiccator (4.2.3)

and, after cooling, weigh it to the nearest 0,001 g (mass m_1). Then weigh into the weighing bottle, to the nearest 0,1 g, about 5 g of the crude sample (see 2.2 of ISO/R 802). Place the open weighing bottle and its cover in the electric oven (4.2.2), controlled at 300 ± 10 °C, and maintain at this temperature for 2 h. Remove it from the oven, place it, covered, in the desiccator (4.2.3) and, after cooling, weigh it to the nearest 0,001 g (mass m_2).

The mass of the test portion, dried at 300 °C, is given by the difference $(m_2 - m_1)$.

4.3.2 *Determination*

Introduce the weighing bottle containing the test portion (4.3.1) into the vessel (4.2.4) containing the carbon tetrachloride (4.1.1). Place it in the centre of the perforated plate forming the support. Open the weighing bottle and close the vessel (4.2.4).

Maintain at 25 ± 0,5 °C for 2 h.

At the end of the 2 h, open the vessel, quickly close the weighing bottle and weigh it again to the nearest 0,001 g (mass m_3).

4.3.3 *Blank test*

4.3.3.1 PRINCIPLE

Determination of the mass of air saturated with carbon tetrachloride vapour, contained in the weighing bottle used in the determination, exposed empty and open, under the same conditions as for the determination.

4.3.3.2 PROCEDURE

Empty the weighing bottle after the determination (4.3.2) and place it open, with its cover, in the vessel (4.2.4) containing the carbon tetrachloride, in the centre of the perforated plate forming the support, and close the vessel. Maintain it at 25 ± 0,5 °C for 2 h. At the end of the 2 h, open the vessel, quickly close the weighing bottle and weigh it again to the nearest 0,001 g (mass m_4).

4.4 Expression of results

The carbon tetrachloride adsorption index, expressed as the volume, in millilitres, of adsorbed carbon tetrachloride per 100 g of aluminium oxide dried at 300 °C, is given by the formula :

$$\frac{(m_3 - m_4) - (m_2 - m_1)}{1{,}594\ (m_2 - m_1)} \times 100$$

where

m_1 is the mass, in grams, of the empty weighing bottle, fitted with its cover, after drying at 300 °C;

m_2 is the mass, in grams, of the weighing bottle, fitted with its cover, containing the test portion (4.3.1) after drying at 300 °C;

m_3 is the mass, in grams, of the weighing bottle, fitted with its cover, containing the test portion (4.3.1), after exposure to the atmosphere of carbon tetrachloride;

m_4 is the mass, in grams, of the weighing bottle, fitted with its cover, after exposure empty and open, to the carbon tetrachloride atmosphere;

1,594 is the density, in grams per millilitre, of the carbon tetrachloride at 25 °C.

4.5 Note

Do not place more than one weighing bottle in each adsorption vessel (4.2.4).

5 SECOND METHOD, FOR ALUMINIUM OXIDE OF DENSITY LOWER THAN 3,75 g/ml

5.1 Reagents

5.1.1 Distilled water

5.1.2 Potassium carbonate, solution saturated at 25 °C.

Mix 840 g of anhydrous potassium carbonate (K_2CO_3) with 600 ml of water (5.1.1) and warm to 40 °C to render completely soluble. The solution partly crystallizes at 25 °C.

5.2 Apparatus

5.2.1 Electric furnace, capable of being controlled at 1 000 to 1 100 °C.

5.2.2 Platinum dish, diameter approximately 75 mm, height approximately 30 mm, provided with a platinum lid.

5.2.3 Desiccator, containing either pure concentrated sulphuric acid or phosphorus pentoxide.

5.2.4 Electric oven, with natural draught, capable of being controlled at 300 ± 10 °C.

5.2.5 Humidity cabinet, 100 % relative humidity atmosphere.

Enclosure fitted with a device for jetting or spraying water on the walls, maintained at 25 ± 5 °C, the test portion being protected against the jets of water.

5.2.6 Desiccator, 44 % relative humidity atmosphere.

Conventional desiccator the lower part of which is three-quarters filled with the potassium carbonate solution (5.1.2), poured when warm into the desiccator.

5.2.7 Enclosure, capable of being controlled at 25 ± 1 °C.

Cabinet with thermally insulated walls, capable of being controlled at 25 ± 1 °C by means of a thermostat, large enough to hold the desiccator (5.2.6) fitted with its cover.

5.3 Procedure

5.3.1 *Test portion*

Heat the platinum dish (5.2.2), fitted with its lid, for 15 min in the electric furnace (5.2.1) controlled at 1 000 to 1 100 °C. Remove it from the furnace, place it in the desiccator (5.2.3) and, after cooling, weigh it to the nearest 0,001 g (mass m_1).

Weigh into the dish, to the nearest 0,01 g, about 2 g of crude sample (see 2.2 of ISO/R 802). Spread the test portion as evenly as possible on the bottom of the dish. Place the open dish, with its lid, in the electric oven (5.2.4), controlled at 300 ± 10 °C, and maintain at this temperature for 2 h. Remove the dish from the oven and place it, covered, in the desiccator (5.2.3). After cooling, weigh the closed dish to the nearest 0,001 g (mass m_2).

The mass of the test portion dried at 300 °C is given by the difference $(m_2 - m_1)$.

5.3.2 *Determination*

Place the uncovered dish and its contents in the humidity cabinet (5.2.5), 100 % relative humidity atmosphere, for 30 min and then transfer it, uncovered, to the desiccator (5.2.6), 44 % relative humidity atmosphere, and leave it there for 1 h 45 min.

Never place more than two test portions in the desiccator (5.2.6) at the same time.

Remove the dish and its contents from the desiccator (5.2.6), put its lid in place and then weigh it to the nearest 0,001 g (mass m_3).

Place the dish, with its contents and its lid, uncovered, for 15 min in an oven controlled at about 110 °C.

Cover the dish and its contents then first heat at 500 to 600 °C for 15 min in the electric furnace (5.2.1), and then at 1 000 to 1 100 °C for 1 h.

Allow the covered dish and its contents to cool to ambient temperature in the desiccator (5.2.3) and weigh it, with its lid, to the nearest 0,001 g (mass m_4).

5.4 Expression of results

The water vapour adsorption index, expressed as the mass, in grams, of water adsorbed per 100 g of aluminium oxide, dried at 300 °C, in an atmosphere of 44 % relative humidity, is given by the formula :

$$\frac{m_3 - m_4}{m_2 - m_1} \times 100$$

where

m_1 is the mass, in grams, of the empty dish, fitted with its lid, after drying at 1 000 to 1 100 °C;

m_2 is the mass, in grams, of the dish, fitted with its lid containing the test portion (5.3.1), after drying at 300 °C;

m_3 is the mass, in grams, of the dish, fitted with its lid, containing the test portion (5.3.1), after exposure in a 44 % relative humidity atmosphere;

m_4 is the mass, in grams, of the dish, fitted with its lid, containing the test portion, after heating at 1 000 to 1 100 °C.

6 TEST REPORT

The test report shall include the following particulars :

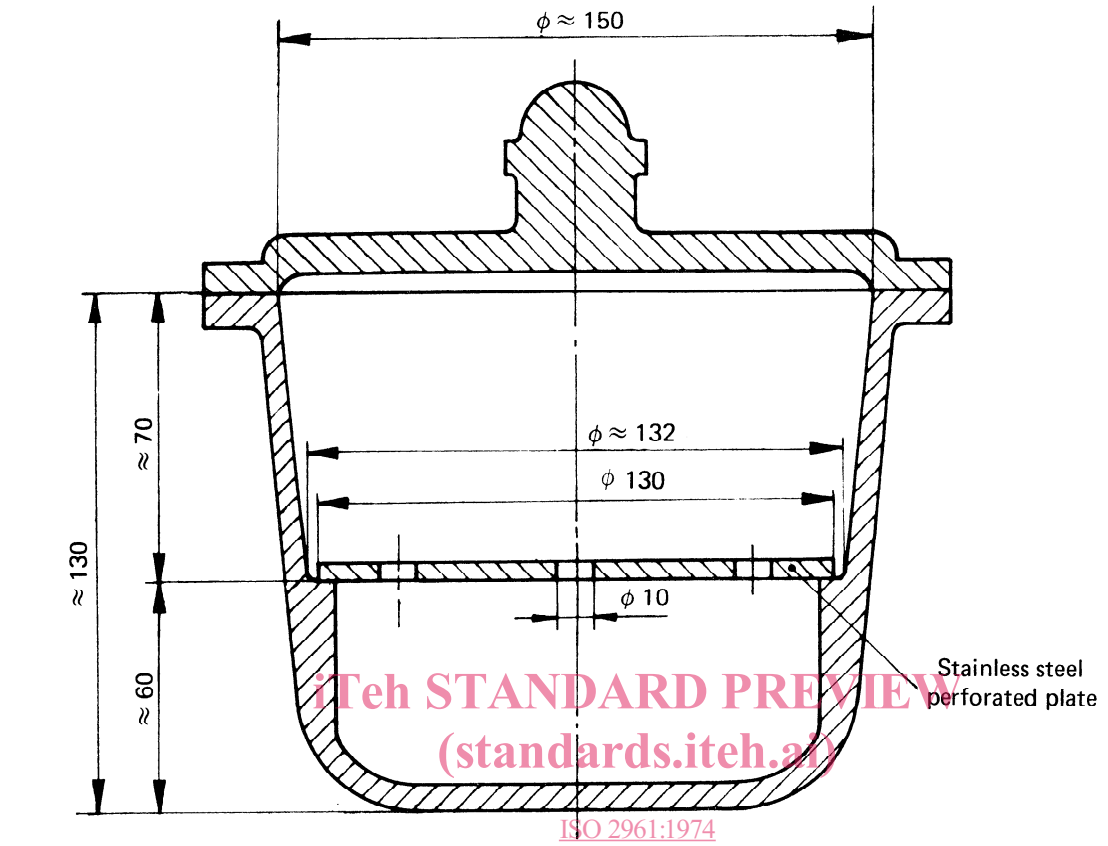
a) the reference of the method used;

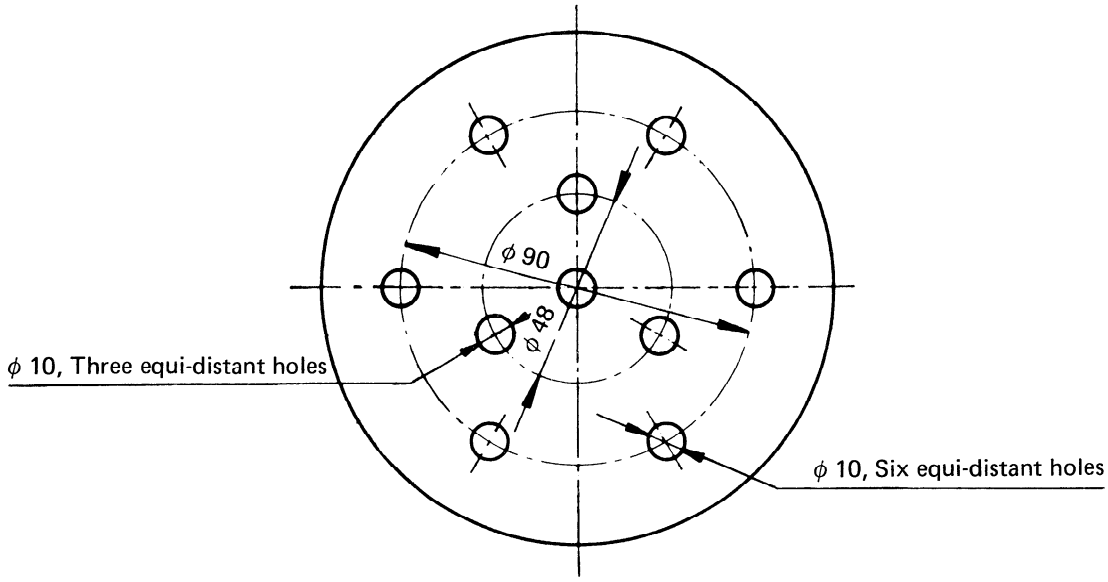
b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard of the document to which reference is made, or regarded as optional.

Dimensions in millimetres





Details of the plate

FIGURE — **Glass vessel for use as a desiccator (4.2.4)**

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