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Aluminium oxide primarily used for the production of aluminium — Determination of boron content — Curcumin spectrophotometric method

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FOREWORD

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International Standard ISO 2865 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in August 1972.

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

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Belgium	Israel	Sweden
Czechoslovakia	Italy	Switzerland
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The Member Body of the following country expressed disapproval of the document on technical grounds :

Netherlands

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Aluminium oxide primarily used for the production of aluminium — Determination of boron content — Curcumin spectrophotometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a curcumin spectrophotometric method for the determination of the boron content of aluminium oxide primarily used for the production of aluminium.

The method is applicable to boron contents, expressed as boron oxide (B_2O_3), greater than 0,000 6 % (i.e. 0,000 2 % expressed as boron (B)).

NOTE — The sensitivity of the method, as may be gathered from the calibration curve, is in fact higher. In the case of a very low blank reading, and with sufficiently sensitive apparatus, the lower limit of application of the method may be extended to 0,000 08 % of boron oxide (B_2O_3), corresponding to 0,000 025 % of boron (B).

2 REFERENCE

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

3 PRINCIPLE

Dissolution of a test portion in phosphoric acid. Separation of the boron by distillation as methyl borate. Formation of a red-coloured complex between the boron and the curcumin reagent at pH 7.

Spectrophotometric measurement of the complex at a wavelength of about 550 nm.

4 REAGENTS

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

4.1 Orthophosphoric acid, ρ approximately 1,70 g/ml, about 85 % (*m/m*) solution.

4.2 Methanol, ρ approximately 0,79 g/ml.

4.3 Ethanol, ρ approximately 0,81 g/ml.

4.4 Glycerol, alkaline solution.

Dissolve 1 g of sodium hydroxide and 0,1 g of sodium chloride in about 100 ml of water to which has been added 3,0 ml of glycerol. Store this solution in a silica flask.

4.5 Curcumin reagent solution.

Add to a 250 ml silica one-mark volumetric flask :

- 175 ml of the ethanol (4.3);
- 3,75 g of oxalic acid [$(COOH)_2.2H_2O$];
- 6,25 ml of hydrochloric acid solution, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution;
- 19 ml of water;
- 0,087 5 g of curcumin.

Shake until completely dissolved, dilute to the mark with the ethanol (4.3) and mix.

Store in the dark and at about 20 °C. This solution is stable for at least 2 months.

4.6 Boron, standard solution, corresponding to 0,08 g of B_2O_3 per litre.

Weigh, to the nearest 0,000 1 g, 0,142 1 g of boric acid (H_3BO_3). Transfer to a silica beaker of convenient capacity (for example 200 ml) and dissolve in water. Transfer the solution quantitatively to a 1 000 ml silica one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution corresponds to 0,08 mg of B_2O_3.

4.7 Boron, standard solution, corresponding to 0,000 8 g of B_2O_3 per litre.

Place 10,0 ml of the standard boron solution (4.6) in a 1 000 ml silica one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution corresponds to 0,8 μg of B_2O_3.

Prepare this solution just before use.

4.8 Thymol blue solution, 0,5 g/l.

Dissolve 0,05 g of thymol blue in water and dilute to 100 ml.

5 APPARATUS

Ordinary laboratory apparatus and :

5.1 Silica apparatus, for the boron distillation.

The apparatus (an example of which is shown in Figures 1 and 2) comprises :

5.1.1 *Distillation flask,* 250 ml with a ground neck;

NOTE — The flask shall be replaced as soon as it shows signs of attack by phosphoric acid.

5.1.2 *Guard tube,* bent and fitted with ground joints;

5.1.3 *Adaptor,* with three ground joints;

5.1.4 *Cylindrical dropping funnel,* with ground joint and stopper;

5.1.5 *Connector,* with two ground joints;

5.1.6 *Liebig condenser,* with ground joints (effective length, about 400 mm).

NOTE — Use different apparatus for the distillation of :

- the blank test;
- test portions of B_2O_3 contents less than 13 μg;
- test portions of B_2O_3 contents greater than 13 μg.

Carefully wash the apparatus before use by heating 100 ml of an approximately 10 N hydrochloric acid solution to boiling and, after rejecting this solution, distilling 100 ml of the methanol (4.2), acidified with hydrochloric acid.

5.2 Silica beaker, capacity about 250 ml.

5.3 Water-bath, capable of being controlled at 55 ± 1 °C (see figure 3).

5.4 Thermometer, covering the temperature range of 20 to 140 °C.

5.5 Stirring rod, constructed of plastics material.

5.6 Platinum dish, diameter about 75 mm, height about 35 mm.

NOTE — Before use, wash the dish carefully first with an approximately 10 N hydrochloric acid solution and then with the methanol (4.2). Use different dishes for :

- the blank test;
- test portions of B_2O_3 contents less than 13 μg;
- test portions of B_2O_3 contents greater than 13 μg;

5.7 Glycerol or silicone oil bath.

5.8 Spectrophotometer.

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 0,001 g, 1 g of the laboratory sample, dried at 300 °C, prepared as described in clause 2.3 of ISO/R 802.

6.2 Blank test

Carry out at the same time and following the same procedure a blank test in the absence of pure aluminium oxide, using the same quantities of all reagents used in the test.

6.3 Preparation of the calibration curve

6.3.1 *Preparation of standard colorimetric solutions,* referring to spectrophotometric measurements made with an optical path length of 2 cm.

Into a series of five platinum dishes (5.6) add the volumes of the standard boron solution (4.7) indicated in the following table.

Standard boron solution (4.7)	Corresponding mass of B_2O_3
ml	μg
0 *	0
1,0	0,8
4,0	3,2
8,0	6,4
12,0	9,6

* compensation solution.

Add to each dish 3,0 ml of the alkaline glycerol solution (4.4).

NOTE 1 — If 4 ml of the alkaline glycerol solution (4.4) is used for the determination (see 6.4.2), use this volume to establish the calibration curve and for the blank test.

Heat the dishes on the water bath (5.3) until removal of the water is complete. Then heat at 130 °C in an electric oven until the residue is dry.

Add to each dish 5,0 ml of the curcumin reagent solution (4.5), rinsing down the contents by running the solution down the wall.

Develop the colour by heating on the water bath (5.3), to complete evaporation, checking, by means of the thermometer (5.4), that the temperature of the water bath is 55 ± 1 °C.

Continue heating for a further 20 min after evaporation is complete.

NOTE 2 — As the colour reaction is influenced by the humidity of the atmosphere above the dishes, the water level in the bath (5.3) is automatically maintained 15 cm from the top. Separate the dishes from each other by distances approximately equivalent to their diameter.

Remove the dishes from the bath and allow to cool in a desiccator.

Dissolve the residues in 25 ml of the ethanol (4.3), stirring with the plastics rod (5.5). Transfer the solutions quantitatively to 50 ml one-mark volumetric flasks, dilute to the marks with the ethanol (4.3) and mix. Filter the solutions through dry fine grade filter papers direct into dry cells of optical path length 2 cm.

Prepare a new calibration curve each time a fresh batch of the curcumin reagent solution (4.5) is prepared.

6.3.2 *Spectrophotometric measurement*

Carry out the spectrophotometric measurements using the spectrophotometer (5.8) at a wavelength of about 550 nm, after having adjusted the apparatus to zero absorbance against the compensation solution.

6.3.3 *Preparation of the calibration chart*

Plot a graph having, for example, on the abscissae the values, expressed in milligrams, of the masses of B_2O_3 per 50 ml of standard colorimetric solution and the corresponding values of absorbance on the ordinates.

6.4 Determination

6.4.1 *Preparation of the test solution*

Place the test portion (6.1) in the distillation flask (5.1.1) and add 20,0 ml of the phosphoric acid solution (4.1). Introduce a plastics-coated magnetic stirring rod. Place the guard tube (5.1.2) in position (see figure 1) so as to ensure that the solution is not contaminated during the dissolution of the test portion. Carefully heat the phosphoric acid, with stirring, until the test portion is completely dissolved. Allow to cool to between 50 and 60 °C, replace the guard tube by the adaptor (5.1.3) and fit the cylindrical dropping funnel (5.1.4), the connector (5.1.5) and the condenser (5.1.6). (See figure 2.) Add 35,0 ml of the methanol (4.2) through the dropping funnel (5.1.4), mix carefully and note the level of liquid in the distillation flask (5.1.1).

6.4.2 *Distillation*

Place 3,0 ml of the alkaline glycerol solution (4.4) in the beaker (5.2), and add 1 to 3 drops of the thymol blue solution (4.8) and 35,0 ml of water. Place the beaker so that the lower end of the condenser (5.1.6) is immersed in this absorption solution.

Heat the distillation flask containing the test solution (6.4.1) to 100 °C by means of the glycerol or silicone oil bath (5.7). Add, through the dropping funnel (5.1.4), 55,0 ml of the methanol (4.2) in 10 ml portions, the final portion being 15 ml. After each addition, mix the solution by means of the magnetic stirrer, without removing the flask from the heating bath, and distil down to the level previously noted. When the methanol has distilled completely, allow the temperature to rise to between 120 and 130 °C.

The colour of the solution in the beaker should never become yellow; if such is the case add a further 1 ml of the alkaline glycerol solution (4.4). (See 6.3.1 Note 1.) Rinse the condenser (5.1.6) with water and add the rinse to the distillate. The total volume of water added shall be equal to at least half the volume of methanol distilled.

6.4.3 *Colour development*

Transfer the contents of the beaker quantitatively to a platinum dish (5.6). Heat carefully on the water-bath (5.3) controlled at 55 ± 1 °C until removal of the water is complete. Then heat at 130 °C in an electric oven until the residue is dry. Increase the temperature to 600 °C in an electric furnace in order to destroy the thymol blue. Maintain at this temperature until the residue is perfectly white. If the residue starts to melt or does not form a uniform layer on the surface of the dish, redissolve it in some water and dry once again, following the procedure already described.

Add 5,0 ml of the curcumin reagent solution (4.5) to the dish, rinsing down the contents by running the solution down the wall. Develop the colour by heating on the water bath (5.3) to complete evaporation, checking, by means of the thermometer (5.4), that the temperature of the water bath is 55 ± 1 °C. Continue heating for a further 20 min after evaporation is complete. Remove the dish from the bath and allow to cool in a desiccator.

Dissolve the residue in 25 ml of the ethanol (4.3), stirring with the plastics rod (5.5). Transfer the solution quantitatively to a 50 ml one-mark volumetric flask, dilute to the mark with the ethanol (4.3) and mix. Filter the solution through a dry fine-grade filter paper direct into a dry cell of optical path length 2 cm.

6.4.4 *Spectrophotometric measurement*

Carry out the spectrophotometric measurement using the spectrophotometer (5.8) at a wavelength of about 550 nm after having adjusted the apparatus to zero absorbance against the blank test solution.

7 EXPRESSION OF RESULTS

Using the calibration curve (6.3.3), determine the mass of boron oxide corresponding to the value of the spectrophotometric measurement.

The content of boron oxide (B_2O_3) is given, as a percentage by mass, by the formula

$$m \times \frac{100}{1\,000} = \frac{m}{10}$$

where m is the mass, in milligrams, of B_2O_3 found in the test solution.

8 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 or the document to which reference is made, or regarded as optional.

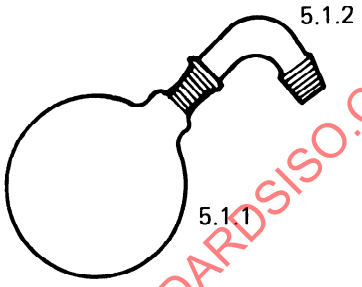

FIGURE 1 — **Assembly for preparation of test solution**

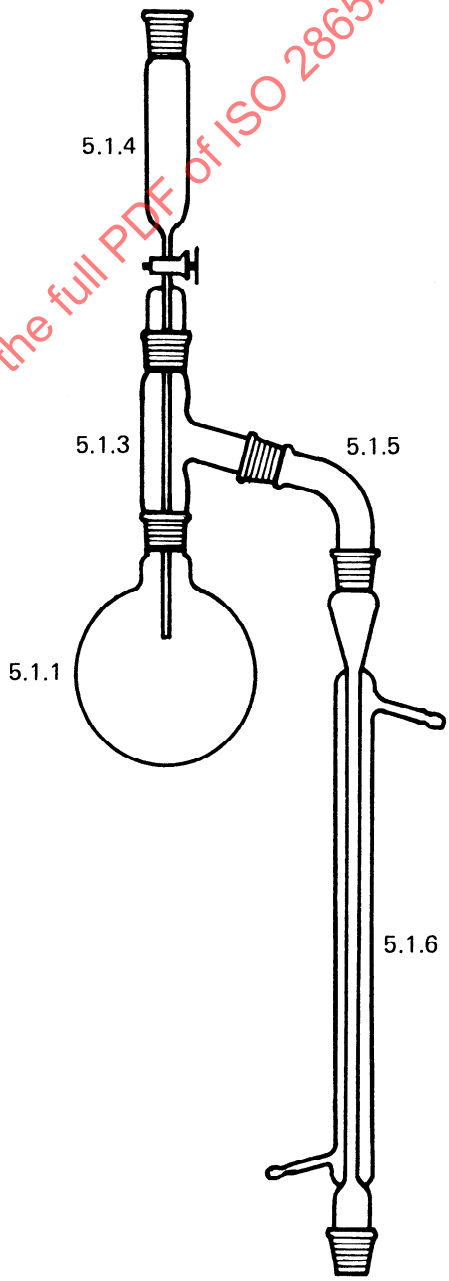

FIGURE 2 — **Assembly for distillation**