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INTERNATIONAL STANDARD 1617

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Aluminium oxide primarily used for the production of aluminium — Determination of sodium content — Flame emission spectrophotometric method

Oxyde d'aluminium principalement utilisé pour la production de l'aluminium — Dosage du sodium — Méthode par spectrophotométrie de flamme en émission

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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 1617 and found it technically suitable for transformation. International Standard ISO 1617 therefore Replaces ISO Recommendation R 1617-1970 to which it is technically identical.

ISO Recommendation R 1617 was approved by the Member Bodies of the following countries :

Australia
Austria
Belgium
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France
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Portugal
Romania
South Africa, Rep. of
Spain
Sweden
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Thailand
Turkey
United Kingdom
U.S.S.R.
Yugoslavia

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 1617 into an International Standard.

Printed in Switzerland

Aluminium oxide primarily used for the production of aluminium — Determination of sodium content — Flame emission spectrophotometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a flame emission spectrophotometric method for the determination of the sodium content of aluminium oxide primarily used for the production of aluminium.

The method is applicable to products having a sodium content, expressed as Na_2O, equal to or greater than 0,05 % *(m/m)*.

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

Fusion, at a controlled temperature, of a test portion of aluminium oxide with a mixture of lithium carbonate and boron trioxide or, as alternative, a mixture of lithium carbonate and lithium tetraborate.

Dissolution of the fused mass in hydrochloric acid.

Aspiration of the solution into a flame and determination of the sodium content by measurement of the intensity of the light emitted at a wavelength of 589 nm.

4 REAGENTS

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

4.1 Lithium carbonate, anhydrous (Li_2CO_3).

4.2 Boron trioxide (B_2O_3), or

4.2.1 Boric acid (H_3BO_3), or

4.2.2 Lithium tetraborate ($Li_2B_4O_7$).

NOTE — If crystallized lithium tetraborate pentahydrate ($Li_2B_4O_7.5H_2O$) is available, first dehydrate it by gradual heating in a platinum dish.

4.3 Aluminium, 99,99 % pure, in the form of shavings.

4.4 Mercury, extra pure.

4.5 Acetone, ρ approximately 0,788 g/ml.

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

4.7 Nitric acid, ρ approximately 1,40 g/ml, about 68 % *(m/m)* solution.

4.8 Sodium, standard solution corresponding to 2,00 g of Na_2O per litre.

Weigh, to the nearest 0,001 g, 3,774 g of sodium chloride, which has been previously dried for about 12 h at 110 °C and cooled in a desiccator, and dissolve in water. Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix. Transfer the solution to a plastics bottle.

1 ml of this standard solution contains 2,00 mg of Na_2O.

4.9 Sodium, standard solution corresponding to 0,200 g of Na_2O per litre.

Take 50,0 ml of the standard sodium solution (4.8) place in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,200 mg of Na_2O.

Prepare this solution just before use and transfer it to a plastics bottle.

4.10 Sodium, standard solution corresponding to 0,080 g of Na_2O per litre.

Take 40,0 ml of the standard sodium solution (4.8), place in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,080 mg of Na_2O.

Prepare this solution just before use and transfer it to a plastics bottle.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Platinum crucible, of upper diameter approximately 50 mm, lower diameter approximately 35 mm and height approximately 40 mm, fitted with a platinum lid, or

5.1.1 Platinum-gold alloy crucible (Au 5 %), having the same dimensions, or

5.1.2 Graphite crucible, of suitable quality and having about the same dimensions as the platinum crucible (5.1).

NOTE – Using a sample of Al_2O_3 of known Na_2O content, a test should be carried out in order to check that the purity and density of the graphite are such as to cause no variation of the Na_2O content. A density of graphite complying with such requirements is equal to or greater than 1,7.

5.2 Open electric furnace, capable of being controlled at 500 ± 20 °C.

5.3 Electric furnace, capable of being controlled at 1 100 ± 50 °C.

5.4 Borosilicate glass beaker, of capacity approximately 400 ml.

5.5 Borosilicate glass beaker, of capacity approximately 600 ml.

5.6 Flame spectrophotometer, provided with an atomizer burner suitably fed to excite the emission of the 589 nm sodium line.

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 0,001 g, 1 g of the test sample, dried at 300 °C (see 3.3 of ISO 802).

6.2 Preparation of the calibration graph

6.2.1 *Preparation of the base solutions*

6.2.1.1 PREPARATION OF AN ALUMINIUM CHLORIDE SOLUTION CORRESPONDING TO 10 g OF Al_2O_3 PER LITRE

Clean about 6 g of the aluminium (4.3) by dipping into a little of the nitric acid solution (4.7). Wash in water and dry with the acetone (4.5). Weigh, to the nearest 0,001 g, 5,294 g of the clean dry metal; place in the beaker (5.5) and add approximately 100 ml of water and 95 ml of the hydrochloric acid solution (4.6). Introduce 1 drop of the mercury (4.4) to facilitate the dissolution. When the reaction subsides, place the beaker on a sand bath and maintain a gentle heat until all the aluminium has dissolved. Allow to cool and transfer the solution to a 1 000 ml one-mark volumetric flask. Wash the beaker and collect the washings in the same flask, dilute to the mark and mix.

Transfer the solution to a plastics bottle.

6.2.1.2 PREPARATION OF A HYDROCHLORIC ACID SOLUTION OF THE FLUX

Place in the beaker (5.4)

- 14 g of the lithium carbonate (4.1) and 17,5 g of the boron trioxide (4.2) or 31 g of the boric acid (4.2.1)

or

- 7,5 g of the lithium carbonate (4.1) and 21 g of the lithium tetraborate (4.2.2).

Add approximately 50 ml of water and, in small portions, 30 ml of the hydrochloric acid solution (4.6). After the liberation of gas has ceased, place the beaker on a sand bath and stir from time to time with a glass rod until the boric acid is completely dissolved. Dilute to approximately 200 ml with hot water, allow to cool and then transfer to a 250 ml one-mark volumetric flask. Wash the beaker and collect the washings in the same flask, dilute to the mark and mix.

Transfer the solution to a plastics bottle.

6.2.2 *Preparation of the standard matching solutions* (see 8.1)

Into a series of eight 100 ml one-mark volumetric flasks, place, in order, 40 ml of the aluminium solution (6.2.1), 10 ml of the flux solution (6.2.1.2) and then the volumes of standard sodium solution (4.9) shown in the following table :

Standard sodium solution (4.9)	Corresponding mass of Na_2O	Mass of Na_2O referred to 100 g of Al_2O_3
ml	mg	g
0*	0	0
5,0	1,00	0,25
10,0	2,00	0,50
12,5	2,50	0,625
15,0	3,00	0,75
17,5	3,50	0,875
20,0	4,00	1,00
25,0	5,00	1,25

* Blank test on reagents for the calibration graph.

Then dilute to the mark and transfer the solutions to plastics bottles.

Use only freshly prepared standard matching solutions.

6.2.3 *Spectrophotometric measurements*

Switch on the spectrophotometer (5.6) a sufficient time in advance to ensure stabilization. Adjust the sensitivity of the apparatus and the slit aperture in accordance with the characteristics of the apparatus used, ensuring a band width of 6 nm at the most, centred on the emission maximum (theoretical value 589 nm).

Aspirate the series of standard matching solutions (6.2.2) into the flame and measure the intensity of the emission.

Ensure that the rate of aspiration of the solutions into the flame is kept constant during the preparation of the calibration graph.

6.2.4 *Plotting the calibration graph*

Plot a graph having, for example, the masses, in milligrams, of Na_2O contained in 100 ml of the standard matching solutions as abscissae and the corresponding values of intensity of emission decreased by the measured value on the blank on the reagents for the calibration graph, on a logarithmic scale, for example, as ordinates.

6.3 Determination

6.3.1 *Preparation of test solution*

Weigh into the crucible (5.1, 5.1.1 or 5.1.2)

- 1,40 g of the lithium carbonate (4.1) and
- 1,75 g of the boron trioxide (4.2) or 3,10 g of the boric acid (4.2.1);

or

- 0,75 g of the lithium carbonate (4.1) and
- 2,10 g of the lithium tetraborate (4.2.2).

Mix carefully, add the test portion (6.1) and mix the whole quantity carefully, preferably with a platinum spatula. Cover the crucible with its lid and place in the open electric furnace (5.2), controlled at 500 ± 20 °C, and maintain this temperature until melting of the flux occurs.

Transfer the covered crucible to the electric furnace (5.3), controlled at 1 100 ± 50 °C, without allowing the temperature to exceed 1 150 °C, and maintain this temperature until fusion of the aluminium oxide is complete (approximately 90 min).

NOTE – Preliminary grinding of the test portion facilitates melting at a lower temperature. If grinding is carried out, use a corundum mortar (see 3.3 of ISO 802).

Remove the crucible from the furnace and allow to cool in air. Heat it again over the flame of a Bunsen burner and then quickly immerse the base of the crucible in a bath of cold water so as to crack the vitrified mass. Detach the pieces of the fused mass with the aid of a platinum rod, if necessary tapping the walls of the crucible lightly with a spatula, and collect them in the beaker (5.4). Add 7,5 ml of the hydrochloric acid solution (4.6) into the beaker, and 5 ml into the crucible. Heat the crucible gently on a sand bath until the residue is completely dissolved. Transfer the solution to the beaker; rinse the crucible several times with hot water, collecting the washings in the beaker. Cover the beaker with a watch-glass and place it on the sand bath. Simmer gently until the fused mass is completely dissolved. Then remove the beaker from the sand bath and allow to cool. Transfer the solution to a 250 ml one-mark volumetric flask, carefully wash the beaker, collecting the washing water in the same flask, dilute to the mark and mix.

Transfer the solution to a plastics bottle.

6.3.2 *Spectrophotometric measurements*

6.3.2.1 PRELIMINARY MEASUREMENT

Carry out a preliminary measurement as specified in 6.2.3 at the same time as the spectrophotometric measurements on the standard matching solutions (6.2.2).

6.3.2.2 BRACKETING MEASUREMENT

Carry out a second measurement on the test solution by bracketing it between two standard matching solutions which differ by only 0,05 g of Na_2O (referred to 100 g of Al_2O_3). Prepare these standard matching solutions using the volumes of the two base solutions (6.2.1.1 and 6.2.1.2) specified in 6.2.2 with the addition of the appropriate volumes of the standard sodium solution (4.10). These quantities shall not differ by more than 2,5 ml.

6.4 Blank test

6.4.1 *Preparation of the solution*

In the beaker (5.4), place the same quantities of the flux as used for the preparation of the test solution (see 6.3.1). Add approximately 50 ml of water and 3 ml of the hydrochloric acid solution (4.6). Place the beaker on a hot-plate and stir from time to time until dissolution is complete. Continue the heating and stirring in order to eliminate carbon dioxide. Cool and transfer the solution to a 250 ml one-mark volumetric flask, which already contains 100 ml of the aluminium solution (6.2.1.1). Wash out the beaker carefully, collect the washings in the same flask, dilute to the mark and mix.

Transfer the solution to a plastics bottle.

6.4.2 *Spectrophotometric measurements*

Carry out the measurements as specified in 6.2.3 in conjunction with the measurements of the test solution and the standard matching solution specified in 6.3.2.1 and 6.3.2.2.

7 EXPRESSION OF RESULTS

The concentration C, expressed in grams per litre, of the solution actually submitted to the spectrophotometer, is given by the formula

$$C = \left[C_1 + (C_2 - C_1)\frac{E - E_1}{E_2 - E_1} \right] - \left[C_3 + (C_4 - C_3)\frac{E_0 - E_3}{E_4 - E_3} \right]$$

where

C_1 is the concentration, in grams per litre, of the weaker standard matching solution used in the determination;

E_1 is the value of the corresponding measurement;

C_2 is the concentration, in grams per litre, of the stronger standard matching solution used in the determination;

E_2 is the value of the corresponding measurement;

E is the value of the measurement corresponding to the determination;

C_3 is the concentration, in grams per litre, of the weaker standard matching solution used in the blank test;

E_3 is the value of the corresponding measurement;

C_4 is the concentration, in grams per litre, of the stronger standard matching solution used in the blank test;

E_4 is the value of the corresponding measurement;

E_0 is the value of the measurement corresponding to the blank test.

The sodium content, expressed as a percentage by mass of sodium oxide (Na_2O), is given by the formula

$$25 \times C$$

8 NOTES ON PROCEDURE

8.1 Bearing in mind the difference in sensitivity between flame photometric instruments, the concentrations of the standard matching solutions specified in 6.2.2 and of the solutions specified in 6.3.1 and 6.4.1 may, if necessary, be modified to enable the measurement to be made in the range of greatest sensitivity of the equipment used.

8.2 Attention is drawn to the necessity of avoiding prolonged contact between solutions (4.8), (4.9), (4.10), (6.2.1.1), (6.2.1.2), (6.2.2), (6.3.1) and (6.4.1) and the glassware used, so that there is no risk of sodium contamination from the glass.

9 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) all operations not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

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.