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

Aluminium fluoride for industrial use — Determination of sodium content — Flame emission spectrophotometric method

ERRATUM

Page 1

Clause 4, 1st line : Add "recognized" after "of".

Sub-clause 4.2 : Replace "**di-Boron**" by "*di*/**Boron**".

Sub-clause 4.5 : Replace the present text by "**Acetone.**"

Page 2

Sub-clause 6.3.1, lines 11 and 12 : Replace "fluxes are" by "the flux has".

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Aluminium fluoride for industrial use — Determination of sodium content — Flame emission spectrophotometric method

Fluorure d'aluminium à usage industriel — 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.

International Standard ISO 4279 was developed by Technical Committee ISO/TC 47, *Chemistry,* and was circulated to the member bodies in September 1975.

It has been approved by the member bodies of the following countries :

Austria	Italy	Spain
Belgium	Mexico	Sweden
France	Netherlands	Switzerland
Germany	Poland	Thailand
Hungary	Romania	Turkey
India	South Africa, Rep. of	United Kingdom

No member body expressed disapproval of the document.

Printed in Switzerland

Aluminium fluoride for industrial use — 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 fluoride for industrial use.

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

2 REFERENCE

ISO 2925, *Aluminium fluoride for industrial use — Preparation and storage of test samples.*

3 PRINCIPLE

Fusion, at a controlled temperature, of a test portion with a mixture of lithium carbonate and boric oxide or boric acid or, alternatively, with a mixture of lithium carbonate and lithium tetraborate.

Extraction of the fused mass with hydrochloric acid solution.

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

4 REAGENTS

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

4.1 Lithium carbonate, anhydrous (Li_2CO_3).

4.2 di-Boron trioxide (Boric oxide) (B_2O_3) or

4.2.1 Boric acid (H_3BO_3) or

4.2.2 Lithium tetraborate ($Li_2B_4O_7$).

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

4.4 Mercury.

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, 3,772 g of sodium chloride, previously dried for 12 h at 110 °C and cooled in a desiccator. Transfer quantitatively to a 1 000 ml one-mark volumetric flask containing a little water, dissolve, dilute to the mark and mix.

Transfer the solution to a suitable plastics bottle.

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

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

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

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

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

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Platinum or platinum-gold alloy (Au 5 %) crucible, of upper diameter about 70 mm and height about 40 mm, with a lid of the same metal.

5.2 Electric furnace, open, capable of being controlled at 500 ± 20 °C.

5.3 Electric furnace, capable of being controlled at 1 000 ± 50 °C.

5.4 Beaker of borosilicate glass, capacity about 600 ml.

5.5 Flame spectrophotometer, fitted with an atomizer-burner fed so as to excite the emission of the 589 nm spectral line of sodium.

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 0,001 g, 1 g of the dried sample (see ISO 2925, sub-clause 2.3).

6.2 Preparation of the calibration graph

6.2.1 *Preparation of the base solutions*

6.2.1.1 PREPARATION OF A HYDROCHLORIC SOLUTION OF ALUMINIUM CORRESPONDING TO 40 g OF AlF_3 PER LITRE

Clean about 6 g of the aluminium (4.3) in a little of the nitric acid solution (4.7). Wash with water and dry with the acetone (4.5). Weigh, to the nearest 0,001 g, 3,212 g of the clean, dry metal, place in the beaker (5.4) and add about 100 ml of water and 95 ml of the hydrochloric acid solution (4.6). Add 1 drop of the mercury (4.4) to facilitate the reaction. Wait for the reaction to subside and heat gently on a hot-plate until all the aluminium is dissolved. Allow to cool slightly, transfer the solution quantitatively to a 250 ml one-mark volumetric flask, dilute to the mark and mix.

Transfer the solution to a suitable plastics bottle.

6.2.1.2 PREPARATION OF A HYDROCHLORIC SOLUTION OF THE FLUX

For this preparation, use the same flux as for the preparation of the test solution (6.3.1).

Place in the beaker (5.4) :

14,0 g of the lithium carbonate (4.1) and 17,5 g of the boric oxide (4.2);

or 14,0 g of the lithium carbonate (4.1) and 31,0 g of the boric acid (4.2.1);

or 7,5 g of the lithium carbonate (4.1) and 21,0 g of the lithium tetraborate (4.2.2).

Add about 50 ml of water and, in small portions, 30 ml of the hydrochloric acid solution (4.6). When the evolution of gas has ceased, heat on a hot-plate, stirring occasionally with a glass rod until dissolution is complete. Dilute to about 200 ml with hot water, allow to cool, transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Transfer the solution to a plastics bottle.

6.2.2 *Preparation of the standard matching solutions*

In a series of eight 100 ml one-mark volumetric flasks, place 10 ml of the aluminium solution (6.2.1.1), 20 ml of the flux solution (6.2.1.2), and then the volumes of the standard sodium solution (4.9) shown in the following table.

Standard sodium solution (4.9)	Corresponding mass of Na_2O	Corresponding mass of Na_2O referred to 100 g of AlF_3
ml	mg	g
0*	0	0
5,00	1,00	0,25
10,00	2,00	0,50
12,50	2,50	0,625
15,00	3,00	0,75
17,50	3,50	0,875
20,00	4,00	1,00
25,00	5,00	1,25

* Blank test on the reagents used for the calibration curve.

Dilute to the mark, mix and transfer the solutions to suitable plastics bottles.

6.2.3 *Spectrophotometric measurements*

Switch on the spectrophotometer (5.5) and allow it to stabilize. Adjust the sensitivity of the apparatus and the slit aperture according to the characteristics of the apparatus so as to ensure a band width of not more than 6 nm, centred on the emission maximum (theoretical value 589 nm).

Aspirate the series of standard matching solutions (6.2.2) in succession in the flame and measure the intensities of the radiations emitted.

Keep the rate of aspiration constant throughout these measurements.

6.2.4 *Preparation of the calibration graph*

Deduct the intensity of the reagent blank from those of the standard matching solutions. 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 corrected values as ordinates.

6.3 Determination

6.3.1 *Preparation of the test solution*

Weigh in the crucible (5.1) :

1,40 g of the lithium carbonate (4.1) and 1,75 g of the boric oxide (4.2);

or 1,40 g of the lithium carbonate (4.1) and 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).

Add the test portion (6.1) and mix carefully with a platinum or nickel or stainless steel spatula. Cover the crucible with its lid, place it in the electric furnace (5.2), controlled at 500 ± 20 °C, and leave at this temperature until fluxes are fused. Transfer the covered crucible into the furnace (5.3), controlled at 1 100 ± 50 °C (do not exceed 1 150 °C), and leave until fusion is complete.

Remove the crucible from the furnace and allow it to cool in air. Heat over a bunsen flame and then quickly immerse the bottom of the crucible in a cold water bath in order to crack the vitrified mass. Detach the pieces of the fused mass using a platinum or nickel or stainless steel rod, lightly tapping the walls of the crucible with a spatula, if necessary, and collect the pieces in the beaker (5.4). Add 7,5 ml of the hydrochloric acid solution (4.6) to the beaker and 5 ml to the crucible. Heat the crucible gently on a hot-plate until dissolution is complete. Then 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 clock glass and place on a hot-plate. Simmer gently until dissolution is complete and allow to cool. Transfer the solution quantitatively to a 250 ml one-mark volumetric flask, dilute to the mark and mix.

Transfer the solution to a suitable plastics bottle.

6.3.2 *Spectrophotometric measurements*

Carry out the flame emission spectrophotometric measurements following the procedure specified in 6.2.3 at the same time as the measurements on the standard matching solutions (6.2.2).

NOTE – Usually low sodium contents do not necessitate the use of the bracketing method.

6.4 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all the reagents as used for the determination.

Transfer the solution to a suitable plastics bottle.

7 EXPRESSION OF RESULTS

Using the calibration graph (6.2.4), determine the concentrations, in milligrams per 100 ml, of Na_2O in the test solution (6.3.1) and in the blank test solution (6.4).

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

$$(c_1 - c_0) \times \frac{1}{1\,000} \times \frac{250}{100} \times \frac{100}{m}$$

$$= \frac{c_1 - c_0}{4\,m}$$

where

c_0 is the concentration, in milligrams per 100 ml, of Na_2O found in the blank test solution (6.4);

c_1 is the concentration, in milligrams per 100 ml, of Na_2O found in the test solution (6.3.1);

m is the mass, in grams, of the test portion (6.1).

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 International Standard to which reference is made, or regarded as optional.

ANNEX

ISO PUBLICATIONS RELATING TO ALUMINIUM FLUORIDE FOR INDUSTRIAL USE

ISO 2362 – Determination of fluorine content – Modified Willard-Winter method.

ISO 2368 – Determination of iron content – 1,10-Phenanthroline photometric method.

ISO 2369 – Determination of silica content – Spectrophotometric method using the reduced silicomolybdic complex.

ISO 2925 – Preparation and storage of test samples.

ISO 3392 – Determination of water content – Electrometric method.

ISO 3393 – Determination of moisture content – Gravimetric method.

ISO 4279 – Determination of sodium content – Flame emission spectrophotometric method.

ISO 4280 – Determination of sulphate content – Barium sulphate gravimetric method.

ISO 5930 – Determination of phosphorus content – Reduced molybdophosphate spectrophotometric method.

ISO 5938 – Determination of sulphur content – X-ray fluorescence method.

ISO . . . – Sampling.

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