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Aluminium oxide primarily used for the production of aluminium — Determination of fluorine content — Alizarin complexone and lanthanum chloride spectrophotometric method

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FOREWORD

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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 2828 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in July 1972.

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

Australia
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Sweden
Switzerland
Thailand
Turkey
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This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

No Member Body expressed disapproval of the document.

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Aluminium oxide primarily used for the production of aluminium — Determination of fluorine content — Alizarin complexone and lanthanum chloride spectrophotometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a spectrophotometric method, using alizarin complexone and lanthanum chloride, for the determination of the fluorine content of aluminium oxide primarily used for the production of aluminium.

The method is applicable to fluorine (F) contents greater than 0,003 %.

2 REFERENCES

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

ISO/R 1693, *Cryolite (natural and artificial) — Determination of fluorine content — Modified Willard-Winter method.*

ISO/R 2073, *Aluminium oxide primarily used for the production of aluminium — Preparation of sample solution for analysis by means of attack by hydrochloric acid under pressure.*

3 PRINCIPLE

Dissolution of a test portion by attack by sulphuric acid under pressure.

Separation of the fluorine by distillation. Formation of a blue-coloured complex between the fluorine and the combined alizarin complexone/lanthanum chloride reagent at a pH between 4,5 ,and 4,6. Addition of acetone to increase the stability and sensitivity of the fluorine complex.

Spectrophotometric measurement of the complex at a wavelength of about 620 nm.

4 REAGENTS

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

4.1 Sulphuric acid, approximately 17,5 N solution.

Carefully add, in small portions, 50 ml of sulphuric acid, ρ approximately 1,84 g/ml, about 96 % (*m*/*m*) solution, to about 50 ml of water and, after cooling, dilute to 100 ml.

4.2 Sulphuric acid, approximately 24 N solution.

Carefully add, in small portions, 200 ml of sulphuric acid, ρ approximately 1,84 g/ml, about 96 % (*m*/*m*) solution, to 100 ml of water and, after cooling, dilute to 300 ml.

4.3 Combined reagent

To prepare this reagent mix, in order, the solutions 4.3.1, 4.3.2 and 4.3.3, add 250 ml of the acetone (4.4) and then dilute to 1 000 ml.

4.3.1 Alizarin complexone solution

Dissolve 0,154 g of alizarin complexone (1,2-dihydroxyanthraquino-3-yl methylamine-*NN*-diacetic acid) in about 100 ml of water to which has been added 1 ml of ammonia solution (ρ approximately 0,91 g/ml, about 24 % (*m*/*m*) solution). After dissolution, add 1 ml of glacial acetic acid (ρ approximately 1,05 g/ml, about 17,4 N solution).

4.3.2 Buffer solution

Dissolve 34 g of sodium acetate trihydrate ($CH_3.COONa.3H_2O$) and 15 ml of glacial acetic acid (ρ approximately 1,05 g/ml, about 17,4 N solution), in about 150 ml of water.

4.3.3 Lanthanum chloride solution

Dissolve 0,372 g of lanthanum chloride heptahydrate ($LaCl_3.7H_2O$) in about 100 ml of water.

NOTE — After mixing the solutions, allow to stand for at least 24 h and, preferably, 2 to 3 days before use. The mixed solution is stable for about 2 weeks. The pH is between 4,55 and 4,60.

4.4 Acetone, ρ approximately 0,80 g/ml.

4.5 Fluorine, standard solution corresponding to 0,200 g of fluorine (F) per litre.

Weigh, to the nearest 0,000 1 g, 0,442 0 g of extra pure anhydrous sodium fluoride (NaF) previously heated to 600 °C in a platinum crucible and allowed to cool in a desiccator. Transfer to a beaker of suitable capacity (for example, 100 ml) and dissolve in water. Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

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

Store the solution in a plastics flask.

NOTE — If extra pure sodium fluoride is not available, recrystallize the product. Dissolve about 5 g of pure sodium fluoride in 125 ml of water and, after dissolution, filter under vacuum through a small Buchner funnel. Then evaporate the solution, in a platinum dish, down to approximately 60 ml.

Cool to about 50 °C and separate the sodium fluoride crystals by centrifuging. Wash the crystals three times, always by centrifuging, with small quantities of cold water.

Transfer the product to a platinum dish and dry in an electric oven, with natural draught, at 110 ± 2 °C.

Remove the dish from the oven, cool in a desiccator, grind the product in an agate mortar, and then pass it through a sieve with a mesh size of 355 µm (see ISO/R 565). Put the sieved sodium fluoride in a platinum dish, heat for 2 h at 600 °C, and allow to cool in a desiccator.

4.6 Fluorine, standard solution corresponding to 0,010 g of fluorine (F) per litre.

Transfer 50,0 ml of the standard solution (4.5) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 10 µg of F.

Prepare this solution immediately before use.

4.7 Fluorine, standard solution corresponding to 0,002 0 g of fluorine (F) per litre.

Transfer 50,0 ml of the standard solution (4.6) to a 250 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 2 µg of F.

Prepare this solution immediately before use.

5 APPARATUS

Ordinary laboratory apparatus, and

5.1 Apparatus specified in ISO/R 2073.

5.2 Apparatus for the steam distillation of fluorine, as specified in 5.3, 5.4 and 5.5 of ISO/R 1693.

5.3 Burette, graduated in 0,05 ml divisions (see ISO/R 385).

5.4 Spectrophotometer.

6 PROCEDURE

6.1 Test portion

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

The mass of test portion should be reduced to 0,5 g if the expected fluorine content is higher than 0,1 %.

6.2 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 used in the test, but omitting the pure aluminium oxide.

6.3 Preparation of the calibration curve

6.3.1 *Preparation of standard colorimetric solutions,* corresponding to measurements carried out with an optical path length of 1 cm.

Into a series of nine 100 ml one-mark volumetric flasks, place the volumes of the standard fluorine solution (4.7) indicated in the following table :

Standard fluorine solution (4.7)	Corresponding mass of fluorine
ml	µg
0*	0
1,0	2
2,0	4
4,0	8
5,0	10
10,0	20
15,0	30
20,0	40
30,0	60

* Compensation solution.

Add to each flask 20 ml of the acetone (4.4) and 40 ml of the combined reagent (4.3).

Check that the pH of each solution is between 4,5 and 4,6, adjusting if necessary by the addition of glacial acetic acid or a 500 g/l ammonium acetate solution.

Dilute the contents of each flask to the mark and mix.

Allow the flasks to stand for 1 h protected from light.

6.3.2 *Spectrophotometric measurements*

After 1 h, carry out the spectrophotometric measurements using the spectrophotometer (5.4) at a wavelength of about 620 nm, after having adjusted the instrument to zero absorbance against the compensation solution.

6.3.3 *Preparation of the calibration chart*

Plot a graph having, for example, the numbers of milligrams of fluorine contained in 100 ml of the standard colorimetric solutions as abscissae and the corresponding values of the absorbance as ordinates.

6.4 Determination

6.4.1 *Preparation of the test solution*

Prepare the test solution using the apparatus specified in 5.1, following the technique in a sealed tube described in ISO/R 2073, substituting, in all cases, 5,5 ml of the sulphuric acid solution (4.1) and 5 ml of water for the specified quantities of hydrochloric acid and water.

If the mass of the test portion is 0,5 g, however, the volumes of sulphuric acid solution (4.1) and water shall be 3 ml and 2,5 ml respectively.

6.4.2 *Distillation*

Place the test solution (6.4.1) in the distillation flask of the apparatus (5.2). Then carry out the distillation procedure by the method described in 6.3.2 of ISO/R 1693, but with the following details :

- distillation with sulphuric acid : use 65 ml of the sulphuric acid solution (4.2);
- temperature of distillation : 150 ± 1 °C.

6.4.3 *Colour development*

Take an aliquot portion of the distillate containing between 2 μg and 60 μg of fluorine, of which the volume shall not exceed 30 ml, and transfer it to a 100 ml one-mark volumetric flask. Add 20 ml of the acetone (4.4) and 40 ml of the combined reagent (4.3).

Check that the pH is between 4,5 and 4,6, adjusting, if necessary, by the addition of glacial acetic acid or a 500 g/l ammonium acetate solution.

Dilute to the mark and mix.

Allow to stand for 1 h protected from light.

6.4.4 *Spectrophotometric measurement*

After 1 h, carry out the spectrophotometric measurement using the spectrophotometer (5.4) at a wavelength of about 620 nm, after having adjusted the instrument to zero absorbance against the blank test solution (6.2).

7 EXPRESSION OF RESULTS

By means of the calibration chart (6.3.3), determine the quantity of fluorine corresponding to the value of the absorbance.

The fluorine content, expressed as a percentage by mass of fluorine (F), is given by the formula :

$$m_1 \times \frac{D}{10 \times m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in milligrams, of fluorine found in the aliquot portion of the test solution;

D is the ratio between the volume of the distillate and the volume of the aliquot portion taken for the colour development.

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 those documents to which reference is made, or regarded as optional.