
INTERNATIONAL STANDARD 2828

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

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.

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-dihydroxy-anthraquino-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 ($\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$) 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 ($\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$) 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.