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Sodium fluoride primarily used for the production of aluminium — Determination of silica content — Reduced molybdosilicate spectrophotometric method

Fluorure de sodium principalemenr utilisé pour la production de l'aluminium — Dosage de la silice — Méthode spectrophotométrique au molybdosilicate réduit

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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 3430 was drawn up by Technical Committee ISO/TC 47, *Chemistry,* and was circulated to the Member Bodies in February 1974.

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

Austria	India	Sweden
Belgium	Ireland	Switzerland
Bulgaria	Israel	Thailand
Chile	Italy	Turkey
Egypt, Arab Rep. of	Poland	United Kingdom
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No Member Body expressed disapproval of the document.

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Sodium fluoride primarily used for the production of aluminium — Determination of silica content — Reduced molybdosilicate spectrophotometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a spectrophotometric method, using the reduced molybdosilicate, for determining the silica content of sodium fluoride primarily used for the production of aluminium.

The method is applicable to products of which the P_2O_5 content does not exceed 0,02 % (*m/m*).

2 REFERENCE

ISO 3428, *Sodium fluoride for industrial use — Preparation and storage of test samples.*

3 PRINCIPLE

Alkaline fusion of a test portion by means of a mixture of sodium carbonate and boric acid. Solution of the fused mass in excess nitric acid so that the pH of the final solution is between 0,3 and 0,5 after being diluted to 250 ml.

Formation, in a suitable aliquot portion, of the oxidized molybdosilicate (yellow) under well-defined conditions of acidity, concentration of reagents, temperature and time.

Selective reduction of the complex in a high acidity sulphuric medium and in the presence of tartaric acid to eliminate interference from phosphorus. Disturbing influence of fluorine is eliminated by the presence of boric acid.

Spectrophotometric measurement of the coloured complex at a wavelength of maximum absorption (about 815 nm).

4 REAGENTS

During the analysis, use only reagents of recognized analytical grade, and only redistilled water.

4.1 Sodium carbonate, anhydrous.

4.2 Boric acid (H_3BO_3).

4.3 Nitric acid, approximately 8 N solution.

Dilute 540 ml of nitric acid solution, ρ approximately 1,40 g/ml, about 68 % (*m/m*) solution, with water to 1 000 ml and mix.

4.4 Sodium molybdate, 195 g/l (0,8 M approximately) solution.

Dissolve, in a beaker made of material free from silica, 19,5 g of sodium molybdate dihydrate ($Na_2MoO_4.2H_2O$) in hot water and, after cooling, dilute to 100 ml and mix. Transfer the solution to a bottle made of material free from silica and, if necessary, filter before use.

4.5 Tartaric acid, 100 g/l solution.

Dissolve 10 g of tartaric acid in water, dilute to 100 ml and mix.

Store the solution in a bottle made of material free from silica.

4.6 Sulphuric acid, approximately 16 N solution.

Carefully add 450 ml of sulphuric acid, ρ approximately 1,84 g/ml, about 96 % (*m/m*) solution, to about 500 ml of water. Cool, dilute to 1 000 ml and mix.

4.7 Reducing solution

Either of the following solutions may be used :

4.7.1 4-amino- 3-hydroxynaphthalene- 1-sulphonic acid, 1,5 g/l solution.

a) Dissolve 7 g of anhydrous sodium sulphite (Na_2SO_3) in 50 ml of water. Add 1,5 g of 4-amino- 3-hydroxynaphthalene- 1-sulphonic acid ($C_{10}H_9NO_4S$);

b) Dissolve 90 g of sodium disulphite ($Na_2S_2O_5$) in 900 ml of water.

Mix the two solutions a) and b), dilute to 1 000 ml and mix. Filter if necessary and store in an amber-coloured bottle made of material free from silica, in a cool place.