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Sodium fluoride primarily used for the production of aluminium — Determination of chlorides content — Turbidimetric method

Fluorure de sodium principalement utilisé pour la production de l'aluminium — Dosage des chlorures — Méthode turbidimétrique

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FOREWORD

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International Standard ISO 3566 was drawn up by Technical Committee ISO/TC 47, Chemistry, and circulated to the Member Bodies in September 1974.

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

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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 chlorides content — Turbidimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a turbidimetric method for the determination of the chlorides content of sodium fluoride primarily used for the production of aluminium.

The method is applicable to products having a chlorides content, expressed as chlorine (CI), equal to or greater than 0.005% (m/m).

2 REFERENCE

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

3 PRINCIPLE

Precipitation, in the presence of boric acid, of chloride ions with silver nitrate and turbidimetric evaluation of the mass of precipitate.

4 REAGENTS

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

- **4.1 Nitric acid**, ρ approximately 1,40 g/ml, about 68 % (m/m) solution.
- **4.2** Boric acid, 40 g/l solution.
- 4.3 Silver nitrate, approximately 0,1 N solution.
- **4.4 Chloride**, standard solution, corresponding to 0,050 g of CI per litre.

Place 14,10 ml of 0,100 N hydrochloric acid solution in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,050 mg of Cl.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Nessler tubes, of capacity 50 ml.

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 0,01 g, 130f the dried test sample. (See ISO 3428, sub-clause 2,30)

6.2 Preparation of the standard matching solutions

Place 12,5 ml of the boric acid solution (4.2) into six of the Nessler tubes (5.1) and then the volumes of the standard chloride solution (4.4) shown in the following table:

Standard chloride solution (4.4)	Corresponding mass of CI
ml	mg
0	0
0,50	0,025
1,00	0,050
2,00	0,100
3,00	0,150
4,00	0,200
5,00	0,250

Dilute to 50 ml, introduce 1,0 ml of the nitric acid solution (4.1) and mix.

6.3 Preparation of the test solution

Place the test portion (6.1) in a 100 ml one-mark volumetric flask and add 60 ml of water and 25 ml of the boric acid solution (4.2). Shake until dissolved, dilute to the mark, and mix. Filter if necessary.

Place 50,0 ml of this solution in one of the Nessler tubes (5.1), add 1,0 ml of the nitric acid solution (4.1) and mix.

6.4 Reaction and turbidimetric evaluation

Add rapidly to the standard matching solutions (6.2) and to the test solution (6.3) 1,0 ml of the silver nitrate solution (4.3), mix and allow to stand in the dark for 5 min.

Compare the opalescence of the test solution with that of each standard matching solution, examining through the axis of the Nessler tubes against a black background and under a diffuse lateral illumination.

Deduce the chlorides content of the test solution

 $\ensuremath{\mathsf{NOTE}}\xspace - \ensuremath{\mathsf{A}}\xspace$ photometer can also be used to measure the degrees of opalescence.