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INTERNATIONAL STANDARD



3566

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

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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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**Descriptors** : sodium fluorides, chemical analysis, determination of content, chlorides, turbidimetric analysis.

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

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

|                |             |                       |
|----------------|-------------|-----------------------|
| Austria        | Hungary     | South Africa, Rep. of |
| Belgium        | India       | Spain                 |
| Bulgaria       | Ireland     | Switzerland           |
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| France         | Netherlands | U.S.S.R.              |
| Germany        | Portugal    | Yugoslavia            |

No Member Body expressed disapproval of the document.

# 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 (Cl), 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**,  $\rho$  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 Cl 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, 1 g of the dried test sample. (See ISO 3428, sub-clause 2.3.)

### 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)<br>ml | Corresponding mass of Cl<br>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

NOTE – A photometer can also be used to measure the degrees of opalescence.

## 7 EXPRESSION OF RESULTS

The chlorides content (Cl) of the dried product, expressed as a percentage by mass, is given by the formula

$$m_1 \times \frac{1}{1\,000} \times \frac{100}{50} \times \frac{100}{m_0} = \frac{m_1}{5 m_0}$$

where

$m_0$  is the mass, in grams, of the test portion (6.1);

$m_1$  is the mass, in milligrams, of chloride found in the test solution.

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

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## ANNEX

### ISO PUBLICATIONS RELATING TO SODIUM FLUORIDE FOR INDUSTRIAL USE AND SODIUM FLUORIDE PRIMARILY USED FOR THE PRODUCTION OF ALUMINIUM

#### SODIUM FLUORIDE FOR INDUSTRIAL USE

ISO 2831 – Determination of water-insoluble matter.

ISO 2832 – Determination of moisture content.

ISO 2833 – Determination of fluorine content – Modified Willard-Winter method.

ISO 3428 – Preparation and storage of test samples.

#### SODIUM FLUORIDE PRIMARILY USED FOR THE PRODUCTION OF ALUMINIUM

ISO 3429 – Determination of iron content – 1,10-Phenanthroline photometric method.

ISO 3430 – Determination of silica content – Reduced molybdosilicate spectrophotometric method.

ISO 3431 – Determination of soluble sulphates content – Turbidimetric method.

ISO 3566 – Determination of chlorides content – Turbidimetric method.

ISO 4278 – Determination of carbonates content – Gravimetric method.