INTERNATIONAL STANDARD 3431

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

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

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

First edition — 1976-07-15

UDC 661.833.316 : 546.226 : 543.436 **Ref. No. ISO 3431-1976 (E)**

Descriptors : chemical compounds, sodium fluorides, chemical analysis, determination of content, sulphates, turbidimetric analysis.

Price based on 3 pages

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 3431 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	Ireland	Sweden
Belgium	Israel	Switzerland
Chile	Italy	Thailand
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The Member Body of the following country expressed disapproval of the document on technical grounds :

Bulgaria

Printed in Switzerland

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

1 SCOPE AND FIELD OF APPLICATION

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

The method is applicable to products of which the soluble sulphates content, expressed as SO_4, is greater than 0,05 % (*m/m*).

2 REFERENCE

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

3 PRINCIPLE

Evaporation of a test portion, in hydrochloric acid solution, to dryness and dissolution of the residue in a hydrochloric acid solution.

Measurement of the turbidity obtained by precipitation, under well-defined conditions, of barium sulphate.

4 REAGENTS

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

4.1 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution or approximately 12 N.

4.2 Barium chloride dihydrate, of uniform particle size between 0,50 and 1,25 mm, standardized by screening.

It is essential that all preparations concerning the determination and standardization shall be carried out using a product of the same particle size distribution.

4.3 Hydrochloric acid, approximately 1 N solution.

4.4 Sulphuric acid, standard solution, corresponding to 0,200 g of SO_4 per litre.

Introduce 41,60 ml of 0,1 N standard volumetric sulphuric acid solution into a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,2 mg of SO_4.

4.5 Glycerol.

4.6 Sodium chloride, 240 g/l acid solution.

Dissolve 120 g of sodium chloride in 450 ml of water in a 500 ml one-mark volumetric flask. Add 10 ml of the hydrochloric acid solution (4.1). Dilute to the mark and mix.

5 APPARATUS

Ordinary laboratory apparatus and :

5.1 Spectrophotometer, or

5.2 Photoelectric absorptiometer, fitted with filters giving only a negligible transmission below 450 nm and above 550 nm.

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 0,01 g, 2 ± 0,1 g of the dried test sample (see ISO 3428, sub-clause 2.3).

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 as used for the determination, but replacing the test portion by 2,5 ml of the standard sulphuric acid solution (4.4), corresponding to 0,5 mg of SO_4, added in order to operate in the linear part of the calibration curve.

6.3 Preparation of calibration graph

6.3.1 *Preparation of the standard matching solutions*

Into each of a series of nine 50 ml one-mark volumetric flasks, place the volumes of the standard sulphuric acid solution (4.4) indicated in the following table.

Standard sulphuric acid solution (4.4)	Corresponding mass of SO_4
ml	mg
0*	0
2,5	0,5
5,0	1,0
7,5	1,5
10,0	2,0
12,5	2,5
15,0	3,0
17,5	3,5
20,0	4,0

* Compensation solution.

Add to each flask 5 ml of the hydrochloric acid solution (4.3), 10 ml of the glycerol (4.5) and 10 ml of the acid sodium chloride solution (4.6), stir, dilute to the mark and mix.

6.3.2 *Turbidimetric reaction*

Transfer 25,0 ml of each standard matching solution (6.3.1), with the exception of the first, to a series of dry 100 ml beakers each containing 0,15 g of the barium chloride (4.2). Swirl by hand for 1 min at a rate of 2 revolutions per second. By this time, the barium chloride should be completely dissolved. Allow to stand for 20 min at a temperature of 20 ± 2 °C.

NOTE — Stagger the tests in order to adhere to the contact times indicated.

6.3.3 *Turbidimetric measurements*

Carry out the turbidimetric measurements using the spectrophotometer (5.1), at a wavelength of about 470 nm, or the photoelectric absorptiometer (5.2), fitted with appropriate filters, after having adjusted the instrument to the optical zero against the compensation solution. Use cells of suitable path length.

NOTE — Stir the solutions by hand before introducing in the cells.

6.3.4 *Preparation of graph*

Plot a graph having, for example, the sulphate contents, expressed as milligrams of SO_4 in the total quantity (50 ml) of the standard matching solutions, as abscissae and the corresponding values of measurements as ordinates.

NOTE — The calibration curve is linear only above 0,5 mg of SO_4.

6.4 Determination

6.4.1 *Preparation of the test solution*

Introduce the test portion (6.1) into a platinum dish of suitable capacity. Add 20 ml of the hydrochloric acid solution (4.1) and evaporate to dryness on a boiling water bath. Repeat this treatment four times.

Dissolve the residue in 5 ml of the hydrochloric acid solution (4.3) and 20 ml of water. Heat for several minutes on a boiling water bath, allow to cool, transfer quantitatively to a 50 ml one-mark volumetric flask, add 2,5 ml of the standard sulphuric acid solution (4.4), corresponding to 0,5 mg of SO_4, added in order to operate in the linear portion of the calibration curve, 10 ml of the glycerol (4.5) and 10 ml of the acid sodium chloride solution (4.6), dilute to the mark and mix. If the solution is turbid, filter on a dry filter collecting the filtrate in a dry receiver.

6.4.2 *Turbidimetric reaction*

Transfer 25,0 ml of the test solution (6.4.1) rapidly to a dry 100 ml beaker containing 0,15 g of the barium chloride (4.2). Swirl by hand, for 1 min, at a rate of 2 revolutions per second. By this time the barium chloride should be completely dissolved. Allow to stand for 20 min at a temperature of 20 ± 2 °C.

6.4.3 *Turbidimetric measurement*

Carry out the turbidimetric measurement by the procedure specified in 6.3.3 after having adjusted the instrument to optical zero against the test solution (6.4.1) and using a cell of the same path length.

NOTE — If the reaction leads to turbidity beyond the limit of the calibration graph, dilute an aliquot portion of the test solution (6.4.1) to 50 ml, after having added the necessary volumes of the glycerol (4.5) and the acid sodium chloride solution (4.6). Then carry out the determination using 25,0 ml of this diluted solution. In this case use this latter as compensation solution and take account of this additional dilution in the calculation of the result.

7 EXPRESSION OF RESULTS

By means of the calibration graph (6.3.4), determine the quantity of sulphate (SO_4) corresponding to the value of the turbidimetric measurement.

The sulphate content, expressed as a percentage by mass of SO_4, is given by the formula

$$(m_1 - m_2) \times \frac{1}{1\,000} \times \frac{100}{m_0} = \frac{m_1 - m_2}{10\,m_0}$$

where

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

m_1 is the mass, in milligrams, of sulphate found in the total quantity (50 ml) of the test solution (6.4.1);

m_2 is the mass, in milligrams, of sulphate found in the total quantity (50 ml) of the blank test solution (6.2).

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.

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.