INTERNATIONAL STANDARD 4280

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

Cryolite, natural and artificial, and aluminium fluoride for industrial use — Determination of sulphate content — Barium sulphate gravimetric method

Cryolithe, naturelle et artificielle, et fluorure d'aluminium à usage industriel — Dosage des sulfates — Méthode gravimétrique à l'état de sulfate de baryum

First edition — 1977-11-15

UDC 661.8 : 553.634 + 661.862.362 : 546.226 : 543.21 **Ref. No. ISO 4280-1977 (E)**

Descriptors : aluminium ores, aluminium fluorides, cryolite, chemical analysis, determination of content, sulphates, gravimetric analysis.

Price based on 3 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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 4280 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in March 1975.

It has been approved by the member bodies of the following countries:

Austria	India	Sweden
Belgium	Israel	Switzerland
Brazil	Italy	Thailand
Bulgaria	Mexico	Turkey
Egypt, Arab Rep. of	Poland	United Kingdom
France	Romania	U.S.A.
Germany	South Africa, Rep. of	U.S.S.R.
Hungary	Spain	

No member body expressed disapproval of the document.

Printed in Switzerland

Cryolite, natural and artificial, and aluminium fluoride for industrial use — Determination of sulphate content — Barium sulphate gravimetric method

1 SCOPE

This International Standard specifies a barium sulphate gravimetric method for the determination of the sulphate content of cryolite (natural and artificial) and aluminium fluoride for industrial use.

2 FIELD OF APPLICATION

The method is applicable to products having sulphate contents, expressed as sulphur trioxide (SO_3), equal to or greater than 0,1 % (*m/m*).

3 REFERENCES

ISO 1619, *Cryolite, natural and artificial — Preparation and storage of test samples.*

ISO 2925, *Aluminium fluoride for industrial use — Preparation and storage of test samples.*

4 PRINCIPLE

Alkaline fusion of a test portion with a mixture of sodium carbonate and boric acid. Extraction of the fused mass with perchloric acid solution and precipitation of the sulphate ions with barium chloride in an acidified medium. Filtration of the precipitate, calcination at 850 °C and weighing of the residue.

5 REAGENTS

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

5.1 Sodium carbonate, anhydrous.

5.2 Boric acid.

5.3 Perchloric acid, ρ approximately 1,67 g/ml, about 70 % (*m/m*) solution.

5.4 Hydrochloric acid, approximately 6 N solution.

5.5 Sulphuric acid, ρ approximately 1,84 g/ml, about 96 % (*m/m*) solution.

5.6 Barium chloride dihydrate ($BaCl_2.2H_2O$), 122 g/l solution, approximately 1 N solution.

5.7 Silver nitrate, 5 g/l nitric acid solution.

Dissolve 0,5 g of silver nitrate in a little water, add 10 ml of nitric acid solution, ρ approximately 1,40 g/ml, dilute to 100 ml with water and mix.

6 APPARATUS

Ordinary laboratory apparatus, and

6.1 Platinum dish, flat bottomed, diameter approximately 75 mm, height approximately 30 mm, with a platinum lid.

6.2 Platinum crucible, diameter approximately 30 mm, height approximately 30 mm.

6.3 Electric oven, capable of being controlled at 110 ± 2 °C.

6.4 Electric furnace, capable of being controlled at 850 ± 20 °C.

7 PROCEDURE

WARNING — Carry out the evaporation of the perchloric acid under a well ventilated fume hood or in a fume cupboard in the absence of ammonia or nitrous vapours.

7.1 Test portion

Weigh, to the nearest 0,000 1 g, in the platinum dish (6.1), about 1 g of the dried test sample (see 3.3 of ISO 1619 or 2.3 of ISO 2925).

7.2 Determination

7.2.1 *Fusion of the test portion*

Add 3,75 g of the sodium carbonate (5.1) and 1,25 g of the boric acid (5.2) to the platinum dish (6.1) containing the test portion (7.1). Mix carefully and cover the dish with the lid.

Place the covered dish in the electric furnace (6.4), controlled at 850 ± 20 °C, and maintain the dish at this temperature for about 30 min. Prevent the dish from touching the furnace shelf by placing it on a suitable support.

7.2.2 *Preparation of the test solution*

Remove the dish from the furnace and allow it to cool in air. Add 25 ml of the perchloric acid solution (5.3) and 15 ml of water and, having replaced the lid, heat until the contents of the dish are completely dissolved. Remove the lid and carefully wash any residue into the dish with hot water. Gently evaporate the contents of the dish to dryness on a hot-plate. Allow to cool. Add 10 ml of the hydrochloric acid solution (5.4). Heat until the residue is completely dissolved and transfer the contents of the dish quantitatively to a 600 ml beaker, washing with hot water until the volume of the solution is about 300 ml.

7.2.3 *Precipitation of sulphates*

Heat the solution to boiling and slowly add, with stirring, 20 ml of the boiling barium chloride solution (5.6). Cover the beaker with a clock-glass and allow the precipitate to settle for about 16 h at ambient temperature.

7.2.4 *Filtration, calcination and weighing*

Filter the settled precipitate on a fine-texture, ashless filter paper (pore diameter approximately 0,4 to 1 μm). Wash the precipitate first by decantation, and transfer it to the filter paper, washing it with boiling water until 10 ml of the filtrate remain clear for 5 min after adding 10 ml of the silver nitrate solution (5.7).

Place the filter paper and its contents in the platinum crucible (6.2), which has been previously heated in the furnace (6.4) at about 850 °C, cooled in a desiccator and weighed. Dry in the electric oven (6.3), controlled at 110 ± 2 °C, for about 1 h, and calcine in the electric furnace (6.4), first at low temperature to avoid burning of the paper, then at 850 ± 20 °C for 30 min. Allow to cool in a desiccator to ambient temperature and, if the calcined precipitate is white, weigh.

If the calcined precipitate appears grey, indicating the presence of graphitic carbon, moisten it with a few drops of the sulphuric acid solution (5.5), replace it in the furnace and calcine at 850 ± 20 °C for about 15 min. Allow to cool in a desiccator to ambient temperature and weigh.

7.3 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 omitting the test portion.

8 EXPRESSION OF RESULTS

The sulphate content, expressed as a percentage by mass of sulphur trioxide (SO_3), is given by the formula

$$\frac{(m_2 - m_1) \times 100 \times 0{,}343\,0}{m_0}$$

$$= \frac{34{,}30\,(m_2 - m_1)}{m_0}$$

where

m_0 is the mass, in grams, of the test portion (7.1);

m_1 is the mass, in grams, of the barium sulphate found in the blank test (7.3);

m_2 is the mass, in grams, of the barium sulphate found in the determination (7.2);

0,343 0 is the factor for conversion of $BaSO_4$ to SO_3.

9 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 Standards to which reference is made, or regarded as optional.

ANNEX

ISO PUBLICATIONS RELATING TO CYROLITE, NATURAL AND ARTIFICIAL, AND ALUMINIUM FLUORIDE FOR INDUSTRIAL USE

CRYOLITE, NATURAL AND ARTIFICIAL

ISO 1619 – Preparation and storage of test samples.

ISO 1620 – Determination of silica content – Reduced molybdosilicate spectrophotometric method.

ISO 1693 – Determination of fluorine content – Modified Willard-Winter method.

ISO 1694 – Determination of iron content – 1,10-Phenanthroline photometric method.

ISO 2366 – Determination of sodium content – Flame emission and atomic absorption spectrophotometric methods.

ISO 2367 – Determination of aluminium content – 8-Hydroxyquinoline gravimetric method.

ISO 2830 – Determination of aluminium content – Atomic absorption method.

ISO 3391 – Determination of calcium content – Flame atomic absorption method.

ISO 3392 – Determination of water content – Electrometric method.

ISO 3393 – Determination of moisture content – Gravimetric method.

ISO 4277 – Conventional test for evaluation of free fluorides content.

ISO 4280 – Determination of sulphate content – Barium sulphate gravimetric method.

ISO 5930 – Determination of phosphorus content – Reduced molybdophosphate spectrophotometric method.

ISO 5938 – Determination of sulphur content – X-ray fluorescence method.

ISO . . . – Sampling.

ALUMINIUM FLUORIDE FOR INDUSTRIAL USE

ISO 2362 – Determination of fluorine content – Modified Willard-Winter method.

ISO 2368 – Determination of iron content – 1,10-Phenanthroline photometric method.

ISO 2369 – Determination of silica content – Spectrophotometric method using the reducec silicomolybdic complex.

ISO 2925 – Preparation and storage of test samples.

ISO 3392 – Determination of water content – Electrometric method.

ISO 3393 – Determination of moisture content – Gravimetric method.

ISO 4279 – Determination of sodium content – Flame emission spectrophotometric method.

ISO 4280 – Determination of sulphate content – Barium sulphate gravimetric method.

ISO 5930 – Determination of phosphorus content – Reduced molybdophosphate spectrophotometric method.

ISO 5938 – Determination of sulphur content – X-ray fluorescence method.

ISO . . . – Sampling.

This page intentionally left blank