

INTERNATIONAL STANDARD

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Acid-grade and ceramic-grade fluorspar — Determination of barium sulfate content — Gravimetric method

*Spathes fluor pour la fabrication de l'acide fluorhydrique et spathes fluor
utilisables dans l'industrie céramique — Dosage du sulfate de baryum —
Méthode gravimétrique*



Reference number
ISO 5437:1992(E)

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 5437 was prepared by Technical Committee ISO/TC 175, *Fluorspar*.

This second edition cancels and replaces the first edition (ISO 5437:1988), which has been updated.

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International Organization for Standardization

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Acid-grade and ceramic-grade fluorspar — Determination of barium sulfate content — Gravimetric method

1 Scope

This International Standard specifies a gravimetric method for the determination of the barium sulfate content of acid-grade and ceramic-grade fluorspar.

The method is applicable to products having barium sulfate contents equal to or greater than 0,1 % (*m/m*).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*.

ISO 8868:1989, *Fluorspar — Sampling and sample preparation*.

3 Principle

Evaporation to dryness of a test portion in the presence of hydrofluoric acid and concentrated sulfuric acid. Extraction of the soluble salts from the residue with a mixture of hydrochloric acid and sulfuric acid.

Filtration, drying and weighing of the residual barium sulfate.

4 Reagents

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

4.1 Hydrofluoric acid, ρ approximately 1,13 g/ml, about 40 g/l solution.

WARNING — Very toxic by inhalation, in contact with skin and if swallowed. Causes severe burns.

Keep container tightly closed in a well-ventilated place. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.

Wear suitable protective clothing and gloves. In case of accident or feeling unwell, seek medical advice immediately (show the label where possible).

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

4.3 Acid mixture.

Add 100 ml of hydrochloric acid [ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution] to 500 ml of water. Then carefully add 20 ml of the sulfuric acid (4.2), mix and dilute to 1 000 ml.

4.4 Ammonium acetate, 200 g/l solution.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Platinum dish, of diameter about 90 mm and capacity about 200 ml.

5.2 Porcelain filter crucible, porosity grade P4 (pore size index 1,6 μm to 4 μm).

5.3 Filter paper, Whatman No. 42 or equivalent.¹⁾

5.4 Platinum crucible.

5.5 Electric oven, capable of being maintained at a temperature of $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

5.6 Electric furnace, capable of being maintained at a temperature of $800\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$.

6 Test sample

Prepare the test sample in accordance with the procedure given in ISO 8868:1989, sub-clause 9.3.

7 Procedure

7.1 Test portion

Grind several grams of the test sample (see clause 6) in an agate mortar until it passes through a 63 μm mesh sieve (see ISO 565). Dry the sieved material for 2 h in the oven (5.5) maintained at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, allow to cool in a desiccator and weigh, to the nearest 0,2 mg, about 1,0 g of this sample into the platinum dish (5.1).

7.2 Determination

Add 20 ml of the hydrofluoric acid (4.1) to the test portion (7.1) in the dish and slowly evaporate to dryness. Add 10 ml of the sulfuric acid (4.2) and evaporate to dryness.

Add 100 ml of the acid mixture (4.3), boil for a few minutes and quantitatively transfer the contents of the dish to a 600 ml beaker. Add 200 ml of the acid mixture to the beaker, insert a glass rod, cover the beaker with a watch glass and boil for 1 h. After boiling, the volume should be about 300 ml.

Allow to stand overnight at ambient temperature and complete the determination in accordance with either of the following procedures.

- a) Filter the contents of the beaker through the porcelain filter crucible (5.2) previously heated in the furnace (5.6), maintained at $800\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$, cooled in a desiccator and weighed to the nearest 0,2 mg. Wash the contents of the porcelain crucible with portions of hot ammonium acetate solution (4.4), using a total volume of 30 ml of the washing reagent.

Carefully wash the contents of the crucible with warm water and heat the crucible for 1 h in the furnace maintained at $800\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$. Allow the crucible to cool to ambient temperature in a desiccator and weigh to the nearest 0,2 mg.

- b) Filter the contents of the beaker through a filter paper (5.3). Wash the filter paper with portions of hot ammonium acetate solution (4.4), using a total volume of 30 ml of the washing reagent. Carefully wash the filter paper with warm water. Transfer the filter paper and residue to the platinum crucible (5.4) which has been previously heated in the furnace (5.6) maintained at $800\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$, cooled in a desiccator and weighed to the nearest 0,2 mg. Heat the crucible in the furnace maintained at $800\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ until the filter paper has been reduced to ashes and allow the crucible to cool to ambient temperature in a desiccator. Moisten the residue in the crucible with a few drops of sulfuric acid (4.2) which has been previously diluted with an equal volume of water.

Reheat the crucible in the furnace, starting from ambient temperature up to $800\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ and hold it at this temperature for 1 h. Allow the crucible to cool to ambient temperature in a desiccator and weigh to the nearest 0,2 mg.

8 Expression of results

The barium sulfate content, expressed as a percentage by mass of BaSO_4 , is given by the formula

$$\frac{m_2 - m_1}{m_0} \times 100$$

where

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

m_1 is the mass, in grams, of the crucible;

m_2 is the mass, in grams, of the crucible and residue.

1) Whatman No. 42 is an example of a suitable filter paper available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this filter paper.

9 Test report

The test report shall include the following particulars:

- a) all information necessary for the identification of the sample;
- b) a reference to the method used (reference to this International Standard);
- c) the results and the form in which they have been expressed;
- d) any unusual features noted during the determination;
- e) any operation not included in this International Standard, or in the International Standards to which reference is made, or regarded as optional.

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