INTERNATIONAL STANDARD

ISO 5437

First edition
1988-12-01

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

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

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

Reference number
ISO 5437 : 1988 (E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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

Printed in Switzerland

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 listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 565 : 1983, *Test sieves — Woven metal wire cloth, perforated plate and electroformed sheet — Nominal sizes of openings.*

ISO 4282 : 1977, *Acid-grade fluorspar — Determination of loss in mass at* 105 °C

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.

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 controlled at 105 °C ± 2 °C.

5.6 Electric furnace, capable of being controlled at 800 °C ± 25 °C.

6 Test sample

Use the residue from the determination of the loss in mass at 105 °C (see ISO 4282) to prepare the test sample.

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.

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) controlled at 105 °C ± 2 °C, allow to cool in a desiccator and weigh, to the nearest 0,2 mg, about 1 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), controlled at 800 °C ± 25 °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 controlled at 800 °C ± 25 °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) controlled at 800 °C ± 25 °C, cooled in a desiccator and weighed to the nearest 0,2 mg. Heat the crucible in the furnace controlled at 800 °C ± 25 °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 °C ± 25 °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.

9 Test report

The test report shall include the following particulars :

a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

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.

UDC 553.634.12 : 543.21 : 546.432'226

Descriptors : minerals and ores, fluorspar, chemical analysis, determination of content, barium sulfate, gravimetric analysis.

Price based on 2 pages