INTERNATIONAL STANDARD

ISO 3703

Second edition
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Acid-grade and ceramic-grade fluorspar — Determination of flotation agents

Spaths fluor pour la fabrication de l'acide fluorhydrique et spaths fluor utilisables dans l'industrie céramique — Détermination des agents de flottation

Reference number
ISO 3703:1993(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 voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 3703 was prepared by Technical Committee ISO/TC 175, *Fluorspar*.

This second edition cancels and replaces the first edition (ISO 3703:1976), which has been technically revised.

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Acid-grade and ceramic-grade fluorspar — Determination of flotation agents

1 Scope

This International Standard specifies a gravimetric method for the determination of the amount of flotation agents adhering to acid-grade and ceramic-grade fluorspar. The method is applicable to materials which have been subjected to a flotation process and which have flotation agent contents equal to or greater than 0,002 % (*m/m*) of the dried material.

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 4282:1992, *Acid-grade and ceramic-grade fluorspar — Determination of loss in mass at 105 °C.*

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

3 Principle

Treatment of a test portion with a mixture of dilute hydrochloric acid and an organic solvent. Removal of the insoluble fluorspar by filtration under vacuum. Separation of the organic phase containing the flotation agent, evaporation of the solvent and weighing of the residue.

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.

4.2 Solvent: 1,1,2-trichlorotrifluoroethane, redistilled.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Vacuum filtration apparatus, comprising a Buchner funnel of diameter 120 mm with suitable filter paper, and a Buchner flask of capacity 1 000 ml.

5.2 Separating funnel, capacity 1 000 ml.

5.3 Mechanical stirrer, powered by an electric motor and fitted with a 40 mm diameter paddle.

6 Test sample

Prepare the test sample in accordance with the procedure given in 9.1 of ISO 8868:1989.

7 Procedure

7.1 Test portion

Weigh into a 1 000 ml beaker, to the nearest 0,1 g, 500 g of the test sample (clause 6). If the loss of mass is required (see 8.2) and has not already been determined according to the method specified for filter cakes in ISO 4282, reserve a portion of the test sample in a closed container for the determination.

7.2 Determination

Add 300 ml of water, 20 ml of the hydrochloric acid solution (4.1) and 200 ml of the solvent (4.2) to the beaker containing the test portion (7.1). Stir vigorously for 30 min using the stirrer (5.3). Filter on the vacuum

filtration apparatus (5.1) and wash the residue with a total volume of 100 ml of the solvent (4.2), added in small portions. Transfer the filtrate, which consists of two phases, from the Buchner flask (5.1) to the separating funnel (5.2) and rinse the flask with a small portion of the solvent (4.2). Draw off the lower phase, passing the liquid through a filter paper in order to remove water from the solvent, and collect it in a flat porcelain dish. Place the dish on a steam bath and evaporate the solvent in a fume cupboard to a volume of a few millilitres[1]. Transfer the residue quantitatively to a beaker, capacity about 50 ml, previously dried at about 100 °C, cooled in a desiccator and weighed to the nearest 0,001 g. Rinse the dish carefully with the solvent (4.2), place the beaker on the steam bath, evaporate to dryness, cool in a desiccator and weigh to the nearest 0,001 g.

8 Expression of results

8.1 The content of flotation agents, expressed as a percentage by mass of the undried material, is given by the formula:

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

where

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

m_1 is the mass, in grams, of the extracted flotation agents.

8.2 The content of flotation agents, expressed as a percentage by mass of the dried material, is given by the formula:

$$\frac{m_1 \times 100 \times f}{m_0}$$

where

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

m_1 is the mass, in grams, of the extracted flotation agents;

f is the correction factor required to express the result on a dry sample basis.

$$f = \frac{100}{100 - L}$$

where L is the loss in mass, expressed as a percentage, as determined by ISO 4282.

9 Test report

The test report shall include the following particulars:

a) all details necessary for the identification of the sample;

b) the method used (a reference to this International Standard);

c) the results and the units in which they have been expressed;

d) any unusual features noted during the determination;

e) details of any operation not included in this International Standard or in the International Standards to which reference is made, as well as details of any operation regarded as optional.

1) Alternatively, for environmental reasons, distill off the solvent in an ordinary distillation apparatus to a volume of a few millilitres. If the solvent is to be re-used, redistill it before use in order to comply with 4.2.

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