INTERNATIONAL STANDARD 4284

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Acid-grade fluorspar — Determination of sulphide content — Iodometric method

Spaths fluor pour la fabrication de l'acide fluorhydrique — Dosage des sulfures — Méthode iodométrique

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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 4284 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in July 1976.

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

Belgium	India	Spain
Brazil	Israel	Switzerland
Bulgaria	Italy	Thailand
Chile	Mexico	Turkey
Czechoslovakia	Netherlands	United Kingdom
France	Poland	Yugoslavia
Germany	Romania	
Hungary	South Africa, Rep. of	

No member body expressed disapproval of the document.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Printed in Switzerland

Acid-grade fluorspar — Determination of sulphide content — Iodometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies an iodometric method for the determination of the sulphide content of acid-grade fluorspar.

The method is applicable to products having a sulphide content equal to or greater than 0,001 % (*m/m*).

NOTE — Acid-grade fluorspar does not normally contain polysulphides. The method is not applicable if their presence is suspected.

2 REFERENCE

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

3 TEST SAMPLE

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

4 PRINCIPLE

Digestion of a test portion in a sealed apparatus in a mixture of hydrochloric acid, tin(II) chloride and boric acid solutions. Absorption of the liberated hydrogen sulphide, entrained in a stream of oxygen-free argon or nitrogen, in cadmium acetate solution and iodometric determination of the cadmium sulphide formed.

5 REAGENTS

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

5.1 Boric acid.

5.2 Nitrogen or argon, oxygen-free.

NOTE — If the presence of oxygen is suspected, first pass the gas through a wash-bottle containing alkaline pyrogallol solution.

5.3 Hydrochloric acid solution, prepared by diluting 1 volume of hydrochloric acid solution, ρ approximately 1,19 g/ml, with 2 volumes of water.

5.4 Tin(II) chloride, 200 g/l solution.

Dissolve 200 g of tin(II) chloride dihydrate ($SnCl_2.2H_2O$) in 300 ml of hydrochloric acid solution, ρ approximately 1,19 g/ml, and dilute with water to 1 000 ml.

5.5 Cadmium acetate, 30 g/l solution.

Dissolve 30 g of cadmium acetate dihydrate [$Cd(CH_3COO)_2.2H_2O$] in water containing 6 ml of glacial acetic acid and dilute to 1 000 ml.

5.6 Iodine, 0,01 N standard volumetric solution.

5.7 Sodium thiosulphate, 0,01 N standard volumetric solution.

NOTE — It is essential that reagents (5.6) and (5.7) should be freshly prepared from 0,1 N standard volumetric solutions.

6 APPARATUS

Ordinary laboratory apparatus and

6.1 Gas evolution and absorption apparatus consisting of

6.1.1 Washing bottle.

6.1.2 Flat bottom flask, fitted with a dropping funnel and a reflux water condenser.

NOTE — A typical apparatus is shown in the figure.

6.2 Electric oven, capable of being controlled at 105 ± 1 °C.

7 PROCEDURE

7.1 Test portion

Grind several grams of the test sample (see clause 3) in an agate mortar until it all passes a 63 µm mesh sieve (see ISO 565). Dry the sieved material for 2 h in the oven (6.2), controlled at 105 ± 1 °C, allow to cool in a desiccator and weigh, to the nearest 0,001 g, about 3 g of this sample.

7.2 Blank test

Carry out a blank test at the same time as the determination, and following the same procedure, using the same reagents as used for the determination but omitting the test portion.

7.3 Determination

Place 50 ml of the cadmium acetate solution (5.5) in the washing bottle (6.1.1). Place the test portion (7.1) in the flask (6.1.2), add 3 g of the boric acid (5.1) and assemble the apparatus (6.1).

Add a mixture of 50 ml of the hydrochloric acid solution (5.3) and 10 ml of the tin(II) chloride solution (5.4) through the dropping funnel.

Insert a one-holed bung fitted with a piece of glass tubing into the neck of the dropping funnel, and pass a stream of the nitrogen or argon (5.2) through the apparatus at a rate of 50 ml/min for 15 min.

Boil the contents of the flask gently for 1 h, without interrupting the gas stream, then disconnect the washing bottle from the apparatus.

Remove the gas inlet tube of the washing bottle and add 10,0 ml of the iodine solution (5.6) and 8 to 10 ml of the hydrochloric acid solution (5.3). Dip the gas inlet tube into the washing bottle, and rinse it carefully, collecting the washings in the bottle. Take great care that all the cadmium sulphide adhering to the inlet tube has been dissolved completely.

Back-titrate the unreacted iodine with the sodium thiosulphate solution (5.7).

8 EXPRESSION OF RESULTS

The sulphide content, expressed as a percentage by mass of sulphur (S), is given by the formula

$$\frac{(10{,}0 - V_1) - (10{,}0 - V_0)}{m} \times 100 \times 0{,}000\ 16$$

$$= \frac{V_0 - V_1}{m} \times 0{,}016$$

where

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

V_0 is the volume, in millilitres, of the sodium thiosulphate solution (5.7) used for the blank test;

V_1 is the volume, in millilitres, of the sodium thiosulphate solution (5.7) used for the determination;

10,0 is the volume, in millilitres, of the iodine solution (5.6) added to the washing bottle;

0,000 16 is the mass, in grams, of sulphur corresponding to 1 ml of exactly 0,01 N sodium thiosulphate solution.

NOTE — If the concentrations of the standard volumetric solutions used are not exactly as specified in the list of reagents, appropriate corrections should be made.

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 Standard to which reference is made, or regarded as optional.

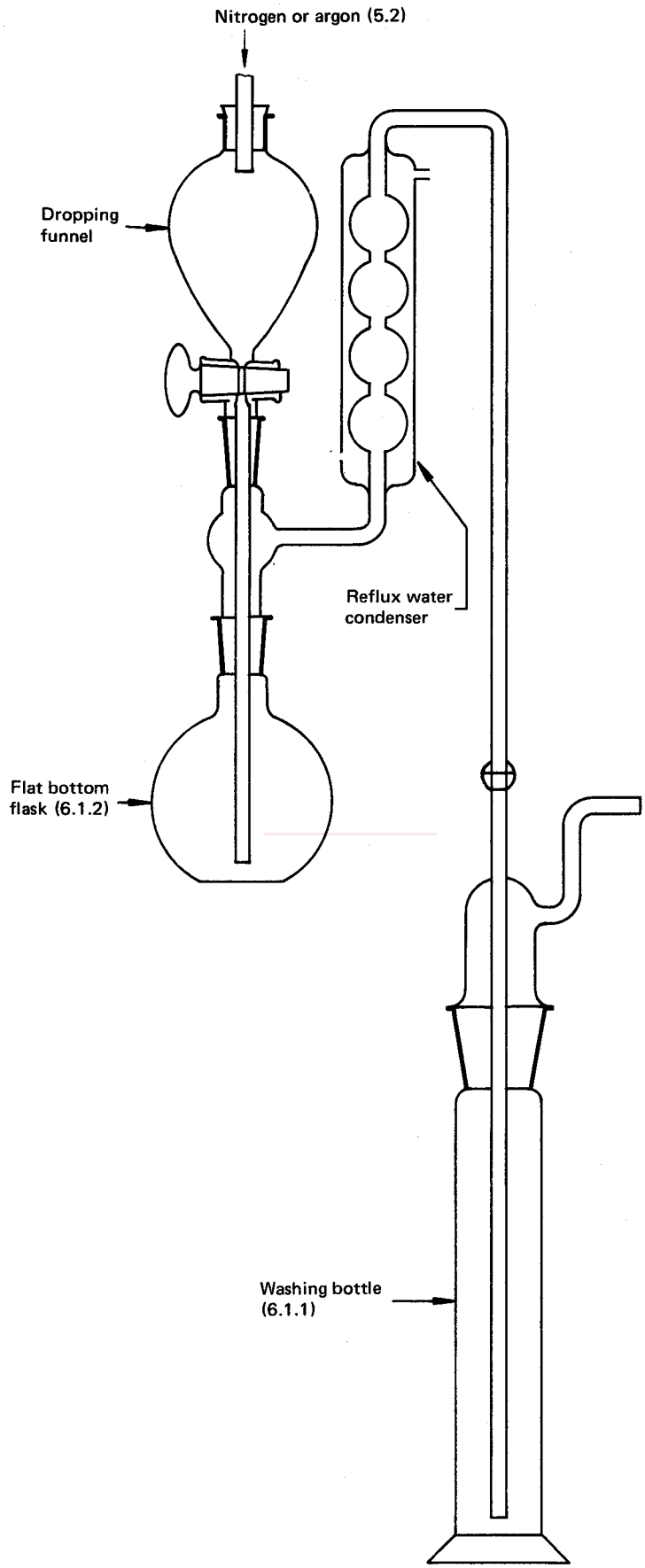

FIGURE – **Typical gas evolution and absorption apparatus**

ANNEX

ISO PUBLICATIONS RELATING TO ACID-GRADE FLUORSPAR

ISO 3703 – Determination of flotation agents.

ISO 4282 – Determination of loss in mass at 105 °C.

ISO 4283 – Determination of carbonate content – Titrimetric method.

ISO 4284 – Determination of sulphides content – Iodometric method.

ISO 5437 – Determination of barium sulphate – Gravimetric method.

ISO 5438 – Determination of silica content – Reduced molybdosilicate photometric method.

ISO 5439 – Determination of available fluorine – Potentiometric method after distillation.