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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

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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).

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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.