INTERNATIONAL STANDARD

ISO 4284

Third edition
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Acid-grade and ceramic-grade fluorspar — Determination of sulfide content — Iodometric method

Spaths fluor pour la fabrication de l'acide fluorhydrique et spaths fluor utilisables dans l'industrie céramique — Dosage des sulfures — Méthode iodométrique

Reference number
ISO 4284:1993(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 4284 was prepared by Technical Committee ISO/TC 175, *Fluorspar*.

This third edition cancels and replaces the second edition (ISO 4284:1988), which has been updated.

Annex A of this International Standard is for information only.

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Acid-grade and ceramic-grade fluorspar — Determination of sulfide content — Iodometric method

1 Scope

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

The method is applicable to products having a sulfide content, expressed as sulfur (S), equal to or greater than 0,001 % (*m*/*m*).

Acid-grade and ceramic-grade fluorspars do not normally contain polysulfides. The method is not applicable if their presence is suspected.

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

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 sulfide, entrained in a stream of oxygen-free argon or nitrogen, in zinc acetate solution and iodometric determination of the zinc sulfide formed.

4 Reagents

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

4.1 Boric acid.

4.2 Nitrogen or **argon**, oxygen-free.

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

4.3 Hydrochloric acid, solution.

Dilute 1 volume of hydrochloric acid (ρ approximately 1,18 g/ml) with 2 volumes of water.

4.4 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 (ρ approximately 1,18 g/ml) and dilute with water to 1 000 ml.

4.5 Zinc acetate, 30 g/l solution.

Dissolve 30 g of zinc acetate dihydrate plus 6 ml of glacial acetic acid in water and dilute to 1 000 ml.

4.6 Iodine, standard volumetric solution, $c(0{,}5\ I_2) = 0{,}005$ mol/l.

It is essential that this solution be freshly prepared by dilution of a standard volumetric solution of iodine, $c(0{,}5\ I_2) = 0{,}05$ mol/l.