

INTERNATIONAL STANDARD

ISO
6676

Third edition
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Acid-grade and ceramic-grade fluorspar — Determination of total phosphorus content — Reduced-molybdophosphate spectrometric method

*Spaths fluor pour la fabrication de l'acide fluorhydrique et spaths fluor
utilisables dans l'industrie céramique — Dosage du phosphore total —
Méthode spectrométrique au molybdophosphate réduit*



Reference number
ISO 6676:1993(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 6676 was prepared by Technical Committee ISO/TC 175, *Fluorspar*.

This third edition cancels and replaces the second edition (ISO 6676:1990), which has been updated.

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Acid-grade and ceramic-grade fluorspar — Determination of total phosphorus content — Reduced-molybdophosphate spectrometric method

1 Scope

This International Standard specifies a reduced-molybdophosphate spectrometric method for the determination of the total phosphorus content of acid-grade and ceramic-grade fluorspar.

The method is applicable to products having total phosphorus contents, expressed as PO_4^{3-} , in the range 0,01 % (m/m) to 1,0 % (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 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

Dissolution of a test portion by fusion with a mixture of sodium carbonate, boric acid and sodium nitrate, and subsequent acidification with nitric acid. Forma-

tion of the molybdophosphate complex and extraction with a mixture of ethyl acetate and butyl acetate, followed by selective reduction of the complex to molybdenum blue by means of tin(II) chloride added to the organic phase.

Spectrometric measurement of the absorbance of the coloured complex at the wavelength of maximum absorption (about 710 nm).

4 Reagents

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

4.1 Sodium carbonate/boric acid, mixture.

Mix 100 g of sodium carbonate and 50 g of boric acid.

4.2 Sodium nitrate.

4.3 Nitric acid, ρ approximately 1,38 g/ml.

4.4 Methanol, ρ approximately 0,794 g/ml.

4.5 Ethyl acetate/butyl acetate, solvent mixture.

Mix 7 volumes of ethyl acetate and 3 volumes of butyl acetate.

4.6 Ammonium molybdate, 30 g/l solution.

Dissolve 30 g of ammonium molybdate tetrahydrate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}]$ and 10 g of ammonium amidosulfate $(\text{NH}_4\text{OSO}_2\text{NH}_2)$ in about 500 ml of water, dilute to 1 000 ml and mix.