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Metallurgical-grade fluorspar — Determination of total phosphorus content — Reduced-molybdophosphate spectrometric method

*Spathes fluor utilisables dans l'industrie métallurgique — Dosage du
phosphore total — Méthode spectrométrique au molybdophosphate
réduit*



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Foreword

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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 9438 was prepared by Technical Committee ISO/TC 175, *Fluorspar*.

This second edition cancels and replaces the first edition (ISO 9438:1989), which has been updated.

Annex A of this International Standard is for information only.

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Metallurgical-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 metallurgical-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

Alkaline fusion of a test portion with a mixture of sodium carbonate, boric acid and sodium nitrate, and subsequent dissolution of the melt in excess of nitric acid. Precipitation of iron(III) phosphate with ammonia solution and filtration, dissolution of the precipitate in nitric acid. Formation 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 Ammonia, approximately 28 % (m/m) solution.

4.7 Ammonia, approximately 0,14 % (m/m) solution.

Dilute 1 volume of ammonia solution (4.6) with 200 volumes of water and mix.

4.8 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.