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Metallurgical-grade fluorspar — Determination of silica content — Reduced-molybdsilicate spectrometric method

*Spaths fluor utilisables dans l'industrie métallurgique — Dosage de la
silice — Méthode spectrométrique au molybdsilicate réduit*



Reference number
ISO 9502:1989(E)

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 9502 was prepared by Technical Committee ISO/TC 175, *Fluorspar*.

Annex A of this International Standard is for information only.

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Metallurgical-grade fluorspar — Determination of silica content — Reduced-molybdosilicate spectrometric method

1 Scope

This International Standard specifies a reduced molybdosilicate spectrometric method for the determination of the silica content of metallurgical-grade fluorspar.

The method is applicable to products having silica contents, expressed as SiO_2 , in the range 0,2 % (m/m) to 30 % (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 565:1983, *Test sieves — Woven metal wire cloth, perforated plate and electroformed sheet — Nominal sizes of openings*.

ISO 4282:1977, *Acid-grade fluorspar — Determination of loss in mass at 105 °C*.

3 Principle

Decomposition of a test portion by fusion with sodium carbonate and subsequent acidification with hydrochloric acid in the presence of boric acid to complex fluoride. Formation of molybdosilicic acid and selective reduction to the blue molybdosilicic acid complex, with addition of tartaric acid to prevent interference from phosphate.

Spectrometric measurement of the absorbance of the coloured complex at a wavelength of approximately 650 nm.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity. All the reagents shall have very low silica contents.

4.1 **Sodium carbonate**, anhydrous.

4.2 **Boric acid**, 40 g/l solution.

4.3 **Hydrochloric acid**, $c(\text{HCl}) \approx 7 \text{ mol/l}$.

4.4 **Sulfuric acid**, $c(0,5\text{H}_2\text{SO}_4) \approx 7 \text{ mol/l}$.

4.5 **Sulfuric acid**, $c(0,5\text{H}_2\text{SO}_4) \approx 18 \text{ mol/l}$.

4.6 **Molybdate**, solution, equivalent to 55 g of Mo per litre, prepared using one of the following procedures.

a) Dissolve 28 g of sodium molybdate dihydrate ($\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$) in 150 ml of water and dilute to 200 ml. Store the solution in a bottle (5.3) and discard if a precipitate appears in the solution.

b) Dissolve 20 g of ammonium molybdate tetrahydrate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$ in 150 ml of water and dilute to 200 ml. Store the solution in a bottle (5.3) and discard if a precipitate appears in the solution.

4.7 **Tartaric acid**, 100 g/l solution.

4.8 **Ascorbic acid**, 20 g/l solution.

Prepare this solution on the day of use.