

# INTERNATIONAL STANDARD

**ISO**  
**5438**

Second edition  
1993-04-01

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## **Acid-grade and ceramic-grade fluorspar — Determination of silica content — Reduced-molybdsilicate spectrometric method**

*Spaths fluor pour la fabrication de l'acide fluorhydrique et spaths fluor  
utilisables dans l'industrie céramique — Dosage de la silice — Méthode  
spectrométrique au molybdsilicate réduit*



Reference number  
ISO 5438:1993(E)

## **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 5438 was prepared by Technical Committee ISO/TC 175, *Fluorspar*.

This second edition cancels and replaces the first edition (ISO 5438:1985), which has been updated.

Annex A of this International Standard is for information only.

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Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

# Acid-grade and ceramic-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 acid-grade and ceramic-grade fluorspar.

The method is applicable to products having silica contents, expressed as  $\text{SiO}_2$ , in the range 0,05 % (m/m) to 4,0 % (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

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 the 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 coloured complex at the wavelength of maximum absorption (about 795 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** solution,  $c(\text{HCl}) \approx 7$  mol/l.

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

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

**4.6 Molybdate** solution, equivalent to 55 g of Mo per litre, prepared by one of the following methods:

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.

or

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.