

INTERNATIONAL STANDARD

ISO 9502

Second edition
1993-04-01

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

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



Reference number
ISO 9502:1993(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 9502 was prepared by Technical Committee ISO/TC 175, *Fluorspar*.

This second edition cancels and replaces the first edition (ISO 9502:1989), which has been updated.

Annex A of this International Standard is for information only.

© ISO 1993

All rights reserved. No part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

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

1 Scope

This International Standard specifies a reduced molybdsilicate 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 565:1990, *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 alkaline fusion with sodium carbonate and subsequent acidification with hydrochloric acid in the presence of boric acid to complex fluoride. Formation of the molybdsilicic acid and selective reduction to the blue molybdsilicic acid complex with addition of tartaric acid to prevent interference from phosphate.

Spectrometric measurement of the absorbance of the coloured complex at a wavelength corresponding to the absorption maximum 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.