

INTERNATIONAL STANDARD

ISO
9505

First edition
1992-01-15

All grades of fluorspar — Determination of arsenic content — Silver diethyldithiocarbamate spectrometric method

*Tous les spaths fluor — Dosage de l'arsenic — Méthode spectrométrique
au diéthylthiocarbamate d'argent*



Reference number
ISO 9505:1992(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 9505 was prepared by Technical Committee ISO/TC 175, *Fluorspar*.

Annex A of this International Standard is for information only.

© ISO 1992

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

All grades of fluorspar — Determination of arsenic content — Silver diethyldithiocarbamate spectrometric method

1 Scope

This International Standard specifies a silver diethyldithiocarbamate spectrometric method for the determination of the arsenic content of all grades of fluorspar.

The method is applicable to products having arsenic (As) contents which are equal to or greater than 0,000 1 % (*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

A test portion is dissolved in a mixture of nitric acid, sulfuric acid, hydrochloric acid and saturated bromine water and evaporated to the stage where the sulfuric acid just commences fuming. The arsenic present is reduced by zinc in a hydrochloric acid medium, to arsine. The arsine is absorbed in a solution of silver diethyldithiocarbamate. The purplish-red colour produced by the colloiddally dispersed silver is measured in a spectrometer at a wavelength of approximately 530 nm.

4 Reagents

All the reagents, and the zinc in particular, shall be free from arsenic or have a very low arsenic content. Only distilled water, or water of equivalent purity, shall be used in the analysis.

4.1 Hydrochloric acid, ρ approximately 1,18 g/ml, about 35 % (*m/m*) solution.

4.2 Nitric acid, ρ approximately 1,38 g/ml, about 60 % (*m/m*) solution.

4.3 Sulfuric acid, diluted 1 + 2, ρ approximately 1,34 g/ml, about 44 % (*m/m*) solution.

Add 1 volume of sulfuric acid, ρ approximately 1,84 g/ml, to 2 volumes of water. Cool the solution while mixing.

4.4 Sulfuric acid, diluted 1 + 10, ρ approximately 1,10 g/ml, about 15 % (*m/m*) solution.

Add 1 volume of sulfuric acid, ρ approximately 1,84 g/ml, to 10 volumes of water. Cool the solution while mixing.

4.5 Sodium hydroxide, approximately 40 g/l solution.

4.6 Bromine water, saturated solution.

4.7 Potassium iodide.

4.8 Tin(II) chloride, hydrochloric acid solution.

Dissolve 40 g of tin(II) chloride dihydrate ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) in hydrochloric acid (4.1) and dilute to 100 ml with the same hydrochloric acid.

Store the solution in an air-tight container.