

INTERNATIONAL
STANDARD

ISO
9779

Second edition
1993-04-01

**Metallurgical-grade fluorspar —
Determination of lead content — Solvent
extraction atomic absorption spectrometric
method**

*Spaths fluor utilisables dans l'industrie métallurgique — Dosage du
plomb — Méthode par spectrométrie d'absorption atomique après
extraction*



Reference number
ISO 9779:1993(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 9779 was prepared by Technical Committee ISO/TC 175, *Fluorspar*.

This second edition cancels and replaces the first edition (ISO 9779:1990), which has been updated.

Annex A of this International Standard is for information only.

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Metallurgical-grade fluorspar — Determination of lead content — Solvent extraction atomic absorption spectrometric method

1 Scope

This International Standard specifies a solvent extraction atomic absorption spectrometric method for the determination of the lead content of metallurgical-grade fluorspar.

The method is applicable to products having lead contents in the range 0,000 6 % (*m/m*) to 0,01 % (*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 decomposed by a mixture of nitric, hydrofluoric and perchloric acids. After evaporation to dryness, the salts are dissolved in hydrochloric acid.

Lead is extracted from a dilute hydrochloric acid solution of the decomposed test portion, containing L(+)-ascorbic acid and sodium iodide, into a solution

of tri-*n*-octylphosphine oxide in 4-methyl-2-pentanone. The 4-methyl-2-pentanone extract is aspirated into the air/acetylene flame of an atomic absorption spectrometer. Spectral energy at 283,3 nm from a lead hollow-cathode lamp is passed through the flame and the absorbance is measured.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 4-methyl-2-pentanone, for use as a spectrometer-zeroing solution.

4.2 Hydrochloric acid, diluted 1 + 1.

Dilute one volume of hydrochloric acid (ρ 1,18 g/ml) with one volume of water.

4.3 Hydrochloric acid, diluted 1 + 9.

Dilute one volume of hydrochloric acid (ρ 1,18 g/ml) with nine volumes of water.

4.4 Nitric acid, ρ 1,38 g/ml.

4.5 Nitric acid, diluted 1 + 1.

Dilute one volume of nitric acid (4.4) with one volume of water.

4.6 Perchloric acid, ρ 1,54 g/ml, about 60 % (*m/m*) solution.

WARNING — Perchloric acid vapour may cause explosions in the presence of ammonia, nitrous fumes or organic matter in general.