

INTERNATIONAL STANDARD

**ISO
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Acid-grade and ceramic-grade fluorspar — Determination of iron content — 1,10-Phenanthroline spectrometric method

*Spaths fluor pour la fabrication de l'acide fluorhydrique et spaths fluor
utilisables dans l'industrie céramique — Dosage du fer — Méthode
spectrométrique à la phénanthroline-1,10*



Reference number
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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 9061 was prepared by Technical Committee ISO/TC 175, *Fluorspar*.

This second edition cancels and replaces the first edition (ISO 9061:1988), which has been updated.

Annex A of this International Standard is for information only.

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Acid-grade and ceramic-grade fluorspar — Determination of iron content — 1,10-Phenanthroline spectrometric method

1 Scope

This International Standard specifies a 1,10-phenanthroline spectrometric method for the determination of the iron content of acid-grade and ceramic-grade fluorspar.

The method is applicable to products having iron contents, expressed as Fe_2O_3 , in the range 0,1 % (m/m) to 2,0 % (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

Alkaline fusion of a test portion with a mixture of sodium carbonate and boric acid. Dissolution of the melt in excess hydrochloric acid. Reduction of the iron(III) with hydroxylammonium chloride. Formation of the iron(II)-1,10-phenanthroline complex in a buffered medium (pH between 3 and 5).

Spectrometric measurement of the coloured complex at a wavelength of about 510 nm, corresponding to the absorption maximum.

4 Reagents

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

4.1 Sodium carbonate, anhydrous.

4.2 Boric acid (H_3BO_3).

4.3 Hydrochloric acid, diluted 1 + 1.

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

4.4 Hydroxylammonium chloride (HONH_3Cl), 10 g/l solution.

4.5 1,10-Phenanthroline monohydrate ($\text{C}_{12}\text{H}_8\text{N}_2 \cdot \text{H}_2\text{O}$), 2 g/l solution.

4.6 Sodium acetate trihydrate ($\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$), 500 g/l solution.

4.7 Iron, standard solution corresponding to 0,100 g of Fe_2O_3 per litre.

Weigh 0,605 g of ammonium iron(III) sulfate 24-hydrate [$\text{Fe}_2(\text{SO}_4)_3 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$] to the nearest 1 mg, place in a beaker and dissolve in water.

Add 10 ml of sulfuric acid (ρ approximately 1,84 g/ml), allow to cool, transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 0,100 mg of Fe_2O_3 .