

INTERNATIONAL STANDARD



5439

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Acid-grade fluorspar — Determination of available fluorine content — Potentiometric method after distillation

*Spathes fluor pour la fabrication de l'acide fluorhydrique — Dosage du fluor utilisable —
Méthode potentiométrique après distillation*

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5439 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in July 1976.

It has been approved by the member bodies of the following countries :

Belgium	Israel	South Africa, Rep. of
Brazil	Italy	Switzerland
Chile	Mexico	Thailand
Czechoslovakia	Netherlands	Turkey
France	Philippines	United Kingdom
Germany	Poland	Yugoslavia
Hungary	Romania	

No member body expressed disapproval of the document.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Acid-grade fluorspar — Determination of available fluorine content — Potentiometric method after distillation

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a potentiometric titration method, using an ion-selective electrode, after distillation, for the determination of the available fluorine content of acid-grade fluorspar.

The method is applicable to products having a calcium fluoride (CaF_2) content equal to or greater than 90 % (m/m).

2 REFERENCES

ISO 2362, *Aluminium fluoride for industrial use — Determination of fluorine content — Modified Willard-Winter method.*

ISO 4282, *Acid-grade fluorspar — Determination of loss in mass at 105 °C.*

3 TEST SAMPLE

Use the residue from the determination of the loss in mass at 105 °C (see ISO 4282) to prepare the test sample.

4 PRINCIPLE

Separation of fluorine from a test portion by distillation in the presence of perchloric acid using a temperature-controlled distillation apparatus. Potentiometric titration of the distillate with lanthanum nitrate solution using a fluoride ion-selective electrode.

5 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled water, or water of equivalent purity. Use carbon dioxide-free water for the preparation of the lanthanum nitrate solution (5.7).

5.1 Potassium permanganate, crystalline.

5.2 Sodium fluoride, recrystallized as follows :

Dissolve about 5 g of sodium fluoride in 125 ml of water and filter the solution under vacuum through a small Buchner funnel. Evaporate the solution in a platinum dish to approximately 60 ml. Cool to about 50 °C and separate

the recrystallized sodium fluoride by centrifuging. Wash the crystals three times by centrifuging, with small quantities of cold water. Transfer the product to a platinum dish and dry in the electric oven (6.9), controlled at 105 ± 2 °C. Remove the dish from the oven, cool in a desiccator, grind the product in an agate mortar, and then pass it through a sieve with an aperture size of 355 μm (see ISO 565). Place the sieved sodium fluoride in a platinum dish, heat for 2 h in an electric furnace controlled at approximately 600 °C and allow to cool in a desiccator.

5.3 Ethanol or propan-2-ol.

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

5.5 Perchloric acid, approximately 10 % (m/m) solution. Dilute 16,5 ml of the perchloric acid solution (5.4) to 100 ml.

5.6 Sodium hydroxide, approximately 1 N solution. Store the solution in a plastic bottle.

5.7 Lanthanum nitrate, approximately 0,01 M standard volumetric solution.

5.7.1 Preparation of the solution

Dissolve 4,33 g of lanthanum nitrate hexahydrate $[\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}]$ in water. Add 10 ml of 0,001 N nitric acid solution, transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

5.7.2 Standardization of the solution

See 7.4.

5.8 Buffer solution, pH 6,5.

Dissolve 79 g of pyridine in about 800 ml of water and neutralize with the perchloric acid solution (5.5) to pH $6,5 \pm 0,2$. Dilute to 1 000 ml with water and, if necessary, readjust the pH to 6,5.

5.9 Phenolphthalein, 5 g/l solution in 95 % (V/V) ethanol.