175

INTERNATIONAL STANDARD

ISO 9061

First edition
1988-12-15

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION
ORGANISATION INTERNATIONALE DE NORMALISATION
МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

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
ISO 9061 : 1988 (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 approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 9061 was prepared by Technical Committee ISO/TC 175, *Fluorspar.*

Annex A of this International Standard is for information only.

Printed in Switzerland

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 listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 565 : 1983, *Test sieves — Woven metal wire cloth, perforated plate and electroformed sheet — Nominal sizes of openings.*

ISO 4282 : 1977, *Acid-grade fluorspar — Determination of loss in mass at* 105 °C.

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.

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 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 ($C_{12}H_8N_2 \cdot H_2O$) 2 g/l solution.

4.6 Sodium acetate trihydrate ($CH_3COONa \cdot 3H_2O$) 500 g/l solution.

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

Weigh, to the nearest 1 mg, 0,605 g of ammonium iron(III) sulfate 24-hydrate [$Fe_2(SO_4)_3(NH_4)_2SO_4 \cdot 24H_2O$], 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.

5 Apparatus

Ordinary laboratory apparatus, and

5.1 Electric oven, capable of being controlled at 105 °C ± 2 °C.

5.2 Platinum dish, flat bottomed, of diameter approximately 80 mm and depth approximately 35 mm, fitted with a platinum lid.

5.3 Muffle furnace, capable of operating at approximately 1 000 °C.

5.4 Molecular absorption spectrometer, fitted with optical cells of optical path length 2 cm.

6 Test sample

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

NOTE — ISO 4282, although specified for acid-grade fluorspar, is equally applicable to ceramic-grade fluorspar.

7 Procedure

7.1 Test portion and preparation of the test solution

Grind several grams of the test sample (clause 6) in an agate mortar until it passes through a 63 µm mesh sieve (see ISO 565). Dry the sieved material for 2 h in the oven (5.1) controlled at 105 ° C ± 2 °C, allow to cool in a desiccator and weigh, to the nearest 1 mg, about 0,5 g of this sample into a platinum dish (5.2).

Add 6 g of the boric acid (4.2) and 4 g of the anhydrous sodium carbonate (4.1) and mix carefully, preferably using a platinum spatula. Cover the dish with its lid, place it on a heat-resistant and thermally insulating plate, heat gently at first and then increase the heat gradually until the reaction slows down. Transfer the dish to the muffle furnace (5.3) operating at approximately 1 000 °C and heat to complete fusion.

Remove the dish from the muffle furnace and allow to cool in air. Add hot water to the dish, heat in a water bath until the melt has dissolved, acidify by adding 20 ml of hydrochloric acid (4.3) slowly and transfer quantitatively to a 250 ml one-mark volumetric flask. Allow to cool, dilute to the mark with water and mix.

7.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all reagents as used for the determination, but omitting the test portion.

7.3 Preparation of the calibration graph

7.3.1 Preparation of the calibration solutions

Into each of a series of seven 250 ml one-mark volumetric flasks, place the volumes of standard iron solution (4.7) shown in table 1.

Table 1

Volume of standard iron solution (4.7) ml	Corresponding mass of Fe_2O_3 mg
0*)	0
1,0	0,1
2,0	0,2
3,0	0,3
4,0	0,4
5,0	0,5
6,0	0,6

*) Compensation solution.

7.3.2 Formation of the absorbing compound

Add to the contents of each volumetric flask an amount of water sufficient to dilute it to approximately 200 ml, then add 5 ml of the hydroxylammonium chloride solution (4.4), mix and allow to stand for 1 min. Add 10 ml of the sodium acetate solution (4.6) and 5 ml of the 1,10-phenanthroline solution (4.5), dilute with water to the mark and mix.

7.3.3 Spectrometric measurements

After 15 min, carry out the spectrometric measurements of the calibration solutions (7.3.1) using the spectrometer (5.4) adjusted to a wavelength of about 510 nm, after having adjusted the instrument to zero absorbance against water.

7.3.4 Plotting the calibration graph

Subtract the absorbance of the calibration compensation solution (see table 1) from the absorbance of each of the calibration solutions to yield the net absorbance.

Plot a calibration graph showing, for example, the mass, in milligrams, of the iron(III) oxide contained in 250 ml of the calibration solutions as abscissa and the corresponding values of net absorbance as ordinate.

7.4 Determination

7.4.1 Aliquot portion of the test solution

In accordance with the estimated iron content, place an aliquot portion, as shown in table 2, of the test solution prepared in 7.1 in a 250 ml one-mark volumetric flask.

Table 2

Fe_2O_3 content % (m/m)	Aliquot portion to be taken ml
0,1 to 0,5	50
0,5 to 1,0	20
1,0 to 2,0	10

7.4.2 Formation of the absorbing compound

To the aliquot portion of the test solution placed in the 250 ml one-mark volumetric flask (7.4.1), add the same quantities of all reagents as used for the standard iron solution (see 7.3.2), dilute to the mark with water and mix.

7.4.3 Spectrometric measurement

After 15 min, carry out the spectrometric measurements of the test solution (7.4.2) and the blank test solution (7.2), following the procedure specified in 7.3.3, after having adjusted the instrument to zero absorbance against water.

8 Expression of results

By reference to the calibration graph (7.3.4), determine the mass of iron corresponding to the values of the absorbances of the test solution and the blank test solution.

The iron content, expressed as a percentage by mass of Fe_2O_3, is given by the formula

$$(m_1 - m_2) \times \frac{r_D}{m_0 \times 10}$$

where

m_0 is the mass, in grams, of the test portion (7.1);

m_1 is the mass, in milligrams, of iron(III) oxide determined in the aliquot portion of the test solution;

m_2 is the mass, in milligrams, of iron(III) oxide determined in the aliquot portion of the blank test solution;

r_D is the ratio of the volume of the test solution to the volume of the aliquot portion taken for the determination.

9 Test report

The test report shall include the following particulars :

a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard, or in the International Standards to which reference is made, or regarded as optional.

Annex A
(informative)

Precision of the method

Comparative analyses carried out in five laboratories on three samples gave the statistical information shown in table A.1.

Table A.1

Sample		1	2	3
Mean of Fe_2O_3 content [% (m/m)]		0,133	0,064	2,07
Standard deviation	of repeatablity, σ_r	0,006	0,006	0,03
	of reproducibility, σ_R	0,008	0,007	0,08

The test samples for the comparative analyses carried out in the five laboratories were distributed to the laboratories after being ground until all the sample passed through a 63 µm mesh sieve.