International Standard 6676

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Acid-grade fluorspar — Determination of total phosphorus content — Molybdophosphate photometric method

Spaths fluor pour la fabrication de l'acide fluorhydrique — Dosage du phosphore total — Méthode photométrique au molybdophosphate

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Foreword

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

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The member body of the following country expressed disapproval of the document on technical grounds :

India

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Acid-grade fluorspar — Determination of total phosphorus content — Molybdophosphate photometric method

WARNING — Attention is drawn to the dangers involved in the use of chloroform and hydrofluoric acid (see the notes to 4.1 and 4.5).

1 Scope and field of application

This International Standard specifies a molybdophosphate photometric method for the determination of the total phosphorus content of acid-grade fluorspar.

The method is applicable to products having total phosphorus contents, expressed as PO_4^{3-}, in the range 0,02 to 1,0 % (m/m).

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2 Reference

ISO 565, *Test sieves — Woven metal wire cloth and perforated plate — Nominal sizes of apertures.*

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

3 Principle

Removal of silica from a test portion by treating first with hydrofluoric acid solution and then with sulphuric acid solution. Dissolution of the residue and preparation of the test solution. Formation of the yellow molybdophosphate complex and photometric measurement of the absorbance of this complex, after extraction with a mixture of butan-1-ol and chloroform, at a wavelength of about 330 nm.

4 Reagents

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

4.1 Butan-1-ol/chloroform, solvent mixture.

Mix equal volumes of the two reagents.

NOTE — Harmful by inhalation. Avoid contact with skin and eyes.

4.2 Nitric acid, ϱ approximately 1,40 g/ml, about 68 % (m/m) solution.

4.3 Nitric acid, approximately 315 g/l solution.

4.4 Sulphuric acid, ϱ approximately 1,84 g/ml, about 96 % (m/m) solution.

4.5 Hydrofluoric acid, ϱ approximately 1,14 g/ml, about 40 % (m/m) solution.

NOTE — Very toxic by inhalation, in contact with skin and if swallowed. Causes severe burns.

Keep container tightly closed in a well-ventilated place. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.

Wear suitable protective clothing and gloves. In case of accident or feeling unwell, seek medical advice immediately (show the label where possible).

4.6 Sodium molybdate dihydrate ($Na_2MoO_4.2H_2O$), approximately 30 g/l solution.

4.7 Phosphorus, standard solution corresponding to 0,100 g of PO_4^{3-} per litre.

Dry a little potassium dihydrogenorthophosphate (KH_2PO_4) by heating in the oven (5.1), controlled at 105 ± 2 °C, for 2 h. Allow to cool in a desiccator. Weigh, to the nearest 0,000 2 g, 0,143 3 g of the dried material, and transfer it quantitatively to a 1 000 ml one-mark volumetric flask. Dissolve in water, dilute to the mark and mix.

1 ml of this standard solution contains 100 µg of PO_4^{3-}.

4.8 Phosphorus, standard solution corresponding to 0,010 g of PO_4^{3-} per litre.

Transfer 100,0 ml of the standard phosphorus solution (4.7) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 10 µg of PO_4^{3-}.

5 Apparatus

Ordinary laboratory apparatus and

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

5.2 Platinum dish, of diameter approximately 90 mm.

5.3 Spectrophotometer, or

5.4 Photometer, fitted with filters providing maximum transmission at a wavelength of about 330 nm.

5.5 Optical cells, made of silica, and of optical path length 1 cm and 4 or 5 cm.

6 Test sample

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

7 Procedure

7.1 Test portion

Grind several grams of the test sample (see clause 6) in an agate mortar until it passes a sieve of aperture size 63 µm (see ISO 565). Dry the ground material for 2 h in the oven (5.1), controlled at 105 ± 2 °C, allow to cool in a desiccator and weigh, to the nearest 0,000 2 g, about 0,1 g into the platinum dish (5.2).

7.2 Blank test

Carry out a blank test simultaneously with 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 calibration graphs

Prepare as follows calibration graphs for the following ranges of phosphorus content, expressed as PO_4^{3-} :

— 0,02 to 0,2 % (*m*/*m*) (calibration graph A);

— 0,2 to 1,0 % (*m*/*m*) (calibration graph B);

7.3.1 Preparation of standard colorimetric solutions

Into each of a series of 500 ml one-mark volumetric flasks, place the volumes of the appropriate standard phosphorus solution (4.7 or 4.8) shown in the following table :

For calibration graph A		For calibration graph B	
Standard phosphorus solution (4.8)	Corresponding mass of PO_4^{3-}	Standard phosphorus solution (4.7)	Corresponding mass of PO_4^{3-}
ml	µg	ml	µg
0*	0	0*	0
2,0	20,0	2,0	200,0
5,0	50,0	4,0	400,0
10,0	100,0	6,0	600,0
15,0	150,0	8,0	800,0
20,0	200,0	10,0	1 000,0

* Blank test on the reagents for calibration.

Treat each of these solutions as follows.

Dilute to approximately 200 ml with water, add 50 ml of the nitric acid solution (4.3), 50 ml of the sodium molybdate solution (4.6), dilute to the mark with water and mix. Transfer a 100,0 ml aliquot portion to a 200 ml separating funnel, add 20 ml of the solvent mixture (4.1) and shake for about 1 min. Allow the layers to separate and filter the lower layer through a filter paper into a 50 ml one-mark volumetric flask. Repeat the extraction and filtration with a further 20 ml portion of the solvent mixture and finally wash the filter paper with a few millilitres of the solvent mixture, collecting the filtrate in the same flask. Dilute to the mark with the solvent mixture and mix.

7.3.2 Photometric measurements

Carry out the measurements using either the spectrophotometer (5.3), at a wavelength of about 330 nm, or with the photometer (5.4) fitted with suitable filters, after having, in each case, adjusted the apparatus to zero absorbance against the solvent mixture (4.1). Use the 4 or 5 cm cells for preparing calibration graph A and the 1 cm cells for preparing calibration graph B.

Deduct the absorbance of the blank test on the reagents for calibration from those of the standard colorimetric solutions (7.3.1).

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7.3.3 Plotting the graphs

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Plot a graph for each range of phosphorus content, having, for example, the masses, expressed in micrograms, of PO_4^{3-} contained in 500 ml of the standard colorimetric solutions (7.3.1) as abscissae, and the corresponding nett values of absorbance as ordinates.

7.4 Determination

7.4.1 Preparation of the test solution

Treat the test portion (7.1) in the platinum dish (5.2) as follows.

Add 10 ml of the hydrofluoric acid solution (4.5), carefully evaporate to dryness on a hot plate in a well-ventilated fume cupboard, and allow to cool. Repeat this operation with a further 10 ml portion of the hydrofluoric acid solution. Add 10 ml of the sulphuric acid solution (4.4) and evaporate until the contents of the dish (5.2) are just moist. Allow to cool.

If necessary, eliminate any organic material present (shown by a brown colour) by adding a few drops of the nitric acid solution (4.2) and heating. Again allow to cool.

Add 50 ml of the nitric acid solution (4.3) and 50 ml of water, heat gently for a few minutes and quantitatively transfer the contents of the dish to a 400 ml beaker. Heat the beaker, boiling gently for about 30 min so as to dissolve the residue. Allow to cool and transfer the solution quantitatively to a 500 ml one-mark volumetric flask. Dilute with about 200 ml of water and then add 50 ml of the sodium molybdate solution (4.6). Dilute to the mark with water and mix. Allow any precipitate present to settle or separate it using a centrifuge.

Transfer a 100,0 ml aliquot portion of this solution to a 200 ml separating funnel and, using the solvent mixture (4.1), carry out the extraction procedure specified in 7.3.1.

7.4.2 Photometric measurements

Using 1 cm cells for PO_4^{3-} contents in the range 0,02 to 0,2 % (m/m) and 4 or 5 cm cells for PO_4^{3-} contents in the range 0,2 to 1,0 % (m/m), carry out the photometric measurements on the extracts obtained from the test solution (7.4.1) and the blank test solution (7.2) following the procedure specified in 7.3.2, after having adjusted the instrument to zero absorbance against the solvent mixture (4.1).

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8 Expression of results

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By means of the appropriate calibration graph, determine the masses of PO_4^{3-} in the test solution and blank test solution, corresponding to the absorbances measured.

The total phosphorus content, expressed as PO_4^{3-} as a percentage by mass, is given by the formula

$$\frac{(m_1 - m_2)}{10^6} \times \frac{100}{m_0}$$

$$= \frac{(m_1 - m_2)}{m_0 \times 10^4}$$

where

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

m_1 is the mass, in micrograms, of PO_4^{3-} found in 500 ml of the test solution (7.4.1);

m_2 is the mass, in micrograms, of PO_4^{3-} found in 500 ml of the blank test solution (7.2).

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 operating details not included in this International Standard or in ISO 4248 to which reference is made, or regarded as optional.

Annex

ISO publications relating to acid-grade fluorspar

ISO 3703 — Determination of flotation agents.

ISO 4282 — Determination of loss in mass at 105 °C.

ISO 4283 — Determination of carbonate content — Titrimetric method.

ISO 4284 — Determination of sulphide content — Iodometric method.

ISO 5437 — Determination of barium sulphate — Gravimetric method.

ISO 5438 — Determination of silica content — Reduced molybdosilicate photometric method.

ISO 5439 — Determination of available fluorine — Potentiometric method after distillation.

ISO 6676 — Determination of total phosphorus content — Molybdophosphate photometric method.

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