International Standard 5930

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Cryolite, natural and artificial, and aluminium fluoride for industrial use — Determination of phosphorus content — Reduced molybdophosphate photometric method

Cryolithe, naturelle et artificielle, et fluorure d'aluminium à usage industriel — Dosage du phosphore — Méthode photométrique au molybdophosphate réduit

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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 :

United Kingdom

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Cryolite, natural and artificial, and aluminium fluoride for industrial use — Determination of phosphorus content — Reduced molybdophosphate photometric method

1 Scope and field of application

This International Standard specifies a reduced molybdophosphate photometric method for the determination of the phosphorus content of natural and artificial cryolite and of aluminium fluoride used primarily for the production of aluminium.

The method is applicable to products having phosphorus contents, expressed as P_2O_5, equal to or greater than 0,002 % (*m/m*).

2 References

ISO 1619, *Cryolite, natural and artificial — Preparation and storage of test samples.*

ISO 2925, *Aluminium fluoride for industrial use — Preparation and storage of test samples.*

3 Principle

Alkaline fusion of a test portion with a mixture of sodium carbonate and boric acid, and dissolution of the fused mass in nitric acid.

Neutralization of a suitable aliquot portion with sodium hydroxide solution. Reaction with acid ammonium molybdate solution at pH 0,3 or below, to form a molybdophosphate complex.

Reduction by a mixture of sodium disulphite and 4-amino-3-hydroxynaphthalene-1-sulphonic acid, and photometric measurement of the reduced complex at a wavelength of about 662 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.

4.3 Nitric acid, approximately 515 g/l solution.

Dilute 540 ml of nitric acid solution, $\varrho \approx$ approximately 1,40 g/ml, about 68 % (*m/m*), with water. Make up the volume to 1 000 ml and mix.

4.4 Ammonium molybdate, 25 g/l acid solution.

Dissolve 25 g of ammonium molybdate tetrahydrate $[(NH_4)_6Mo_7O_{24}.4H_2O]$ in 200 ml of water at about 60 °C. Cool and dilute to 1 000 ml with sulphuric acid, approximately 490 g/l solution.

Store this solution in a bottle made from phosphorus-free material.

4.5 Reducing solution.

Dissolve 1,75 g of sodium sulphite (Na_2SO_3) in 20 ml of water. To this solution, add 0,35 g of 4-amino-3-hydroxynaphthalene-1-sulphonic acid ($C_{10}H_9NO_4S$).

Prepare separately a solution of 22,5 g of anhydrous sodium disulphite ($Na_2S_2O_5$) in 200 ml of water.

Transfer both solutions to a 250 ml one-mark volumetric flask, dilute to the mark and mix.

4.6 Sodium hydroxide, approximately 400 g/l solution.

4.7 Phosphorus, standard solution corresponding to 0,100 g of P_2O_5 per litre.

Weigh, to the nearest 0,000 1 g, 0,194 4 g of sodium dihydrogenphosphate monohydrate ($NaH_2PO_4.H_2O$) and dissolve in water. Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,100 mg of P_2O_5.

4.8 Phosphorus, standard solution corresponding to 0,010 g of P_2O_5 per litre.

Transfer 50 ml of the standard phosphorus solution (4.7) to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,010 mg of P_2O_5.

4.9 Phenolphthalein, 10 g/l solution in 95 % (*V*/*V*) ethanol.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Platinum crucible or **dish,** of base diameter about 60 mm, upper diameter about 80 mm and depth about 35 mm, with a platinum lid.

5.2 Gas burner, capable of achieving a temperature of 800 °C.

5.3 Spectrophotometer, or

5.4 Photoabsorptiometer, fitted with filters providing maximum transmission at a wavelength of about 662 nm.

6 Procedure

6.1 Preparation of the calibration graph

6.1.1 Preparation of the standard colorimetric solutions, for photometric measurements carried out with cells of thickness 4 or 5 cm.

Into a series of six 100 ml one-mark volumetric flasks, place the volumes of the standard phosphorus solution (4.8) specified in the following table :

Standard phosphorus solution (4.8)	Corresponding mass of P_2O_5
ml	mg
0 *	0 *
2,0	0,020
5,0	0,050
10,0	0,100
15,0	0,150
20,0	0,200

* Compensation solution.

6.1.2 Colour development

Treat each of the standard colorimetric solutions (6.1.1) as follows.

Add to each flask 10 ml of the acid ammonium molybdate solution (4.4). Dilute to 80 ml with water and add 2 ml of the reducing solution (4.5). Dilute to the mark and mix. Allow to stand away from light for 30 min.

6.1.3 Photometric measurements

Carry out the photometric measurements using either the spectrophotometer (5.3) at the wavelength of maximum absorption (about 662 nm), or the photoabsorptiometer (5.4) fitted with suitable filters, after having adjusted the apparatus to zero absorbance against water.

6.1.4 Plotting the graph

Deduct the absorbance of the compensation solution (see 6.1.1) from that of each of the standard colorimetric solutions (6.1.1). Plot a graph having, for example, the P_2O_5 contents, expressed in milligrams per 100 ml of standard colorimetric solution, as abscissae, and the corresponding values of absorbance as ordinates.

6.2 Determination

6.2.1 Test portion

Weigh, to the nearest 0,001 g, 2 g of the laboratory sample (see ISO 1619 for cryolite or ISO 2925 for aluminium fluoride), previously dried at 110 °C and ground.

6.2.2 Preparation of the test solution

In the platinum crucible or dish (5.1), carefully mix, using a small platinum spatula, the test portion (6.2.1) with 12 g of the sodium carbonate (4.1) and 4 g of the boric acid (4.2).

Cover the crucible or dish with its lid and heat at approximately 800 °C for 15 min with the gas burner (5.2) under a well ventilated fume hood. Allow to cool. Then add to the crucible or dish, in small portions, 40 ml of the nitric acid solution (4.3). Heat the solution at just below boiling point for a few minutes and transfer the solution quantitatively to a 250 ml one-mark volumetric flask. Allow to cool, dilute to the mark and mix.

6.2.3 Colour development

Using a pipette, take a suitable aliquot portion of the test solution (6.2.2) and place in a 100 ml one-mark volumetric flask. Add 3 drops of the phenolphthalein solution (4.9) and neutralize with the sodium hydroxide solution (4.6). Add 10 ml of the acid ammonium molybdate solution (4.4) and dilute to 80 ml with water; the pH of this solution should be 0,3 or less. Add 2 ml of the reducing solution (4.5), dilute to the mark and mix. Allow to stand away from light for 30 min.

6.2.4 Blank test

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

6.2.5 Photometric measurements

Carry out the photometric measurement on the test solution (6.2.2) and on the blank test solution (6.2.4) as specified in 6.1.3, after having adjusted the apparatus to zero absorbance against water.

7 Expression of results

By means of the calibration graph (6.1.4), determine the mass of P_2O_5 corresponding to the values of the absorbances measured on the test solution and on the blank test solution.

The phosphorus content, expressed as a percentage by mass of phosphorus(V) oxide (P_2O_5), is given by the formula

$$(m_1 - m_2) \times \frac{250}{V} \times \frac{1}{1\,000} \times \frac{100}{m_0}$$

$$= \frac{25\,(m_1 - m_2)}{m_0 V}$$

where

m_0 is the mass, in grams, of the test portion (6.2.1);

m_1 is the mass, in milligrams, of P_2O_5 found in the aliquot portion of the test solution used for the determination;

m_2 is the mass, in milligrams, of P_2O_5 found in the corresponding aliquot portion of the blank test solution;

V is the volume, in millilitres, of the aliquot portion of the test solution used for the determination.

8 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

ISO publications relating to cryolite, natural and artificial, and aluminium fluoride for industrial use

Cryolite, natural and artificial

ISO 1619 — Preparation and storage of test samples.

ISO 1620 — Determination of silica content — Reduced molybdosilicate spectrophotometric method.

ISO 1693 — Determination of fluorine content — Modified Willard-Winter method.

ISO 1694 — Determination of iron content — 1,10-Phenanthroline photometric method.

ISO 2366 — Determination of sodium content — Flame emission and atomic absorption spectrophotometric methods.

ISO 2367 — Determination of aluminium content — 8-Hydroxyquinoline gravimetric method.

ISO 2830 — Determination of aluminium content — Atomic absorption method.

ISO 3391 — Determination of calcium content — Flame atomic absorption method.

ISO 3392 — Determination of water content — Electrometric method.

ISO 3393 — Determination of moisture content — Gravimetric method.

ISO 4277 — Evaluation of free fluorides content — Conventional titrimetric method.

ISO 4280 — Determination of sulphates content — Barium sulphate gravimetric method.

ISO 5930 — Determination of phosphorus content — Reduced molybdophosphate photometric method.

ISO 5938 — Determination of sulphur content — X-ray fluorescence method.

ISO 6374 — Determination of phosphorus content — Atomic absorption spectrometric method.

Aluminium fluoride for industrial use

ISO 2362 — Determination of fluorine content — Modified Willard-Winter method.

ISO 2368 — Determination of iron content — 1,10-Phenanthroline photometric method.

ISO 2369 — Determination of silica content — Spectrophotometric method using the reduced silicomolybdic complex.

ISO 2925 — Preparation and storage of test samples.

ISO 3392 — Determination of water content — Electrometric method.

ISO 3393 — Determination of moisture content — Gravimetric method.

ISO 4279 — Determination of sodium content — Flame emission spectrophotometric method.

ISO 4280 — Determination of sulphates content — Barium sulphate gravimetric method.

ISO 5930 — Determination of phosphorus content — Reduced molybdophosphate photometric method.

ISO 5938 — Determination of sulphur content — X-ray fluorescence method.

ISO 6374 — Determination of phosphorus content — Atomic absorption spectrometric method.

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