INTERNATIONAL STANDARD 5440

G-92-14

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

Sodium hexafluorosilicate for industrial use — Determination of phosphate content — Molybdovanadate spectrophotometric method

Hexafluorosilicate de sodium à usage industriel — Dosage des phosphates — Méthode spectrophotométrique au molybdovanadate

First edition — 1978-10-01

UDC 661.488 : 546.185 : 543.42

Ref. No. ISO 5440-1978 (E)

Descriptors : chemical compounds, sodium fluorosilicates, chemical analysis, determination of content, orthophosphates, spectrophotometric analysis.

Price based on 3 pages

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 5440 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
Brazil
Chile
Czechoslovakia
Egypt, Arab Rep. of
France
Germany
Hungary
India
Israel
Korea, Rep. of
Mexico
Netherlands
Philippines
Poland
Romania
South Africa, Rep. of
Spain
Switzerland
Thailand
Turkey
United Kingdom
U.S.S.R.
Yugoslavia

No member body expressed disapproval of the document.

Printed in Switzerland

Sodium hexafluorosilicate for industrial use — Determination of phosphate content — Molybdovanadate spectrophotometric method

WARNING — Sodium hexafluorosilicate is poisonous if taken internally. Breathing of the dust should be avoided. Contact with the eyes and skin should be prevented and operators should wash thoroughly after handling the material and should wear a respirator and goggles when handling the powdered material.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a molybdovanadate spectrophotometric method for the determination of the phosphate content of sodium hexafluorosilicate for industrial use.

The method is applicable to products having phosphate contents, expressed as P_2O_5, of 0,005 to 0,05 % *(m/m)*.

2 REFERENCE

ISO 5444, *Sodium hexafluorosilicate for industrial use — 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 5444) to prepare the test sample.

4 PRINCIPLE

Dissolution of a test portion in dilute hydrochloric and nitric acid solutions. Formation of the yellow molybdovanadate and spectrophotometric measurement at a wavelength of about 420 nm. Calculation of the phosphate content, expressed as P_2O_5, from the measured absorbance.

NOTE — If the molybdovanadate complex is used instead of the blue molybdate complex, it is not necessary to remove fluorosilicate. There is consequently no need for a reduction step.

5 REAGENTS

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

5.1 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % *(m/m)* solution.

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

5.3 Ammonium molybdovanadate, nitric acid solution.

Dissolve 20 g of ammonium molybdate tetrahydrate $[(NH_4)_2MoO_4.4H_2O]$ in about 500 ml of water, with heating. When dissolved, add 1 g of ammonium metavanadate (NH_4VO_3) and allow to dissolve. Cool the solution, and add, in small portions, with swirling, 150 ml of the nitric acid solution (5.2). Cool the solution, dilute to 1 000 ml with water and mix.

5.4 Phosphate, standard solution, corresponding to 0,10 g of P_2O_5 per litre.

Weigh, to the nearest 0,001 g, 1,92 g of potassium dihydrogen phosphate (KH_2PO_4), dissolve it in water in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

Immediately before use, transfer 10,0 ml of this solution to a 100 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution corresponds to 0,10 mg of P_2O_5.

6 APPARATUS

Ordinary laboratory apparatus and

6.1 Spectrophotometer, fitted with cells of 1 cm optical path length.

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0,001 g, 10 ± 0,1 g of the test sample, dried at 105 °C (see clause 3), into a 250 ml beaker.

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 the reagents, but omitting the test portion.

7.3 Preparation of the test solution

Add 100 ml of water, 10 ml of the hydrochloric acid solution (5.1) and 10 ml of the nitric acid solution (5.2) to the beaker containing the test portion (7.1). Place a clock-glass on the beaker and heat for 10 min to digest the test portion. Allow to cool. In the absence of insoluble material, transfer the solution quantitatively to a 250 ml one-mark volumetric flask. If any insoluble material is present, filter it off, wash the filter repeatedly with water and collect the filtrate and washings in a 250 ml one-mark volumetric flask. Finally, dilute to the mark and mix.

7.4 Preparation of calibration graph

7.4.1 *Preparation of standard colorimetric solutions* for spectrophotometric measurements carried out with cells of 1 cm optical path length

Into a series of eleven 100 ml one-mark volumetric flasks, place the volumes of the standard phosphate solution (5.4) shown in the following table.

Standard phosphate solution (5.4)	Corresponding mass of P_2O_5
ml	mg
0*	0
1,0	0,10
2,0	0,20
3,0	0,30
4,0	0,40
5,0	0,50
6,0	0,60
7,0	0,70
8,0	0,80
9,0	0.90
10,0	1,0

* Compensation solution.

Treat the contents of each flask as follows. Dilute to about 50 ml with water, add 2 ml of the hydrochloric acid solution (5.1) and 2 ml of the nitric acid solution (5.2). Then add 25 ml of the ammonium molybdovanadate solution (5.3) and mix. Dilute to the mark, mix and allow to stand for not less than 10 min.

7.4.2 *Spectrophotometric measurements*

Carry out the spectrophotometric measurements using the spectrophotometer (6.1), at a wavelength of about 420 nm, in 1 cm cells, after having adjusted the instrument to zero absorbance against water.

7.4.3 *Plotting the graph*

Deduct the absorbance of the compensation solution from that of each standard colorimetric solution and plot a graph having, for example, the numbers of milligrams of P_2O_5 contained in 100 ml of standard colorimetric solution as abscissae and the corresponding values of absorbance as ordinates.

7.5 Determination

7.5.1 *Colour development*

Place 50,0 ml of the test solution (7.3) into a 100 ml one-mark volumetric flask, add 25 ml of the ammonium molybdovanadate solution (5.3), dilute to the mark, mix and allow to stand for not less than 10 min.

7.5.2 *Spectrophotometric measurements*

Carry out the spectrophotometric measurements on the test solution and on the blank test solution (7.2), after colour development, by the procedure specified in 7.4.2.

8 EXPRESSION OF RESULTS

By means of the calibration graph (7.4.3), determine the masses of P_2O_5 corresponding to the absorbances of the aliquot portions of the test and blank test solutions taken for colour development.

The phosphate content, expressed as a percentage by mass of P_2O_5, is given by the formula

$$\frac{m_1 - m_2}{1\,000\, m_0} \times 100 \times \frac{250}{50} = \frac{m_1 - m_2}{2\, m_0}$$

where

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

m_1 is the mass, in milligrams, of P_2O_5 found in the aliquot portion of the test solution taken for colour development;

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

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 Standard to which reference is made, or regarded as optional.

ANNEX

ISO PUBLICATIONS RELATING TO SODIUM HEXAFLUOROSILICATE FOR INDUSTRIAL USE

ISO 4281 — Free acidity and sodium hexafluorosilicate content.

ISO 5440 — Determination of phosphate content — Molybdovanadate spectrophotometric method.

ISO 5443 — Determination of iron content — 1,10-Phenanthroline spectrophotometric method.

ISO 5444 — Determination of loss in mass at 105 °C.

ISO 5915 — Determination of particle size distribution — Sieving method.