INTERNATIONAL STANDARD 4281

G-92-14

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

Sodium hexafluorosilicate for industrial use — Determination of free acidity and total hexafluorosilicate content — Titrimetric method

Hexafluorosilicate de sodium à usage industriel — Déterminations de l'acidité libre et de la teneur en hexafluorosilicate total — Méthode titrimétrique

First edition — 1977-03-01

UDC 661.833 : 543.241.5

Ref. No. ISO 4281-1977 (E)

Descriptors : chemical compounds, sodium fluorsilicate, chemical analysis, determination of content, acidity, sodium fluorsilicate, volumetric analysis.

Price based on 2 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 4281 was developed by Technical Committee ISO/TC 47, *Chemistry,* and was circulated to the member bodies in August 1975.

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

Belgium	India	Spain
Brazil	Israel	Switzerland
Czechoslovakia	Italy	Turkey
Egypt, Arab Rep. of	Mexico	United Kingdom
France	Poland	U.S.S.R.
Germany	Romania	
Hungary	South Africa, Rep. of	

No member body expressed disapproval of the document.

Printed in Switzerland

4281-77 4851903 0032236 4

Sodium hexafluorosilicate for industrial use — Determination of free acidity and total hexafluorosilicate content — Titrimetric 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 titrimetric method for the determination of the free acidity and the total hexafluorosilicate content of sodium hexafluorosilicate for industrial use. In respect of free acidity, it is applicable to products having free acidities, expressed as a percentage by mass of hexafluorosilicic acid (H_2SiF_6), equal to or greater than 0,1.

2 PRINCIPLE

Determination of the free acidity by titration of an ice-cold test portion with a standard volumetric sodium hydroxide solution in the presence of potassium nitrate and using bromothymol blue as indicator, followed by titration at near boiling temperature to determine the total hexafluorosilicate content.

3 REAGENTS

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

3.1 Crushed ice, prepared from distilled water or water of equivalent purity.

3.2 Potassium nitrate, saturated solution at ambient temperature.

3.3 Sodium hydroxide, 0,1 N standard volumetric solution.

3.4 Sodium hydroxide, 0,5 N standard volumetric solution.

3.5 Bromothymol blue, 0,4 g/l solution.

Heat 0,1 g of bromothymol blue with 3,2 ml of 0,05 N sodium hydroxide solution and 5 ml of 95 % (*V/V*) ethanol; after solution is effected, add 50 ml of 95 % (*V/V*) ethanol, dilute to 250 ml with water and mix.

4 REACTIONS

Cold titration

$$H_2SiF_6 + 2NaOH \rightarrow Na_2SiF_6 + 2H_2O$$

Hot titration

$$Na_2SiF_6 + 4NaOH \rightarrow 6NaF + SiO_2 + 2H_2O$$

5 APPARATUS

Ordinary laboratory apparatus.

6 PROCEDURE

6.1 Test portion

In a glass weighing bottle, weigh, to the nearest 0,001 g, between 1,00 and 1,20 g of the test sample.

6.2 Determinations

Transfer the test portion (6.1) quantitatively to a 500 ml beaker containing about 100 g of the crushed ice (3.1) and 25 ml of the potassium nitrate solution (3.2), rinsing the residue from the weighing bottle into the beaker with the minimum quantity of water. Wash down the sides of the beaker with water. While stirring constantly, titrate immediately with the standard volumetric sodium hydroxide solution (3.3), in the presence of a few drops of the bromothymol blue solution (3.5) as indicator, until the blue colour persists for at least 30 s. On longer standing, the indicator will turn yellow; this colour change may be ignored.

Add the bulk of the standard volumetric sodium hydroxide solution (3.4) which is likely to be required for the second titration.

Then place the beaker on a hot-plate and bring to the boil. Titrate the hot solution rapidly with the standard volumetric sodium hydroxide solution (3.4) to the appearance of a permanent blue colour.

7 EXPRESSION OF RESULTS

7.1 The free acidity, expressed as a percentage by mass of hydrochloric acid (HCl), is given by the formula

$$V_0 \times 0{,}003\ 65 \times \frac{100}{m} = \frac{0{,}365\ V_0}{m}$$

or, as a percentage by mass of hexafluorosilicic acid (H_2SiF_6), by the formula

$$V_0 \times 0{,}007\ 205 \times \frac{100}{m} = \frac{0{,}720\ 5\ V_0}{m}$$

where

V_0 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.3) used in the first titration;

m is the mass, in grams, of the test portion (6.1);

0,003 65 is the mass in grams, of hydrochloric acid corresponding to 1 ml of exactly 0,1 N sodium hydroxide solution $\left(\text{namely } \frac{HCl}{1\ 000} \times 0{,}1\right)$;

0,007 205 is the mass, in grams, of hexafluorosilicic acid corresponding to 1 ml of exactly 0,1 N sodium hydroxide solution $\left(\text{namely } \frac{H_2SiF_6}{2\ 000} \times 0{,}1\right)$.

NOTE — The results may be expressed in other terms if appropriate to the manufacturing process used.

7.2 The total hexafluorosilicate (SiF_6^{2-}) content, expressed as a percentage by mass of sodium hexafluorosilicate (Na_2SiF_6), is given by the formula

$$V_1 \times 0{,}023\ 51 \times \frac{100}{m} = \frac{2{,}351\ V_1}{m}$$

where

V_1 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.4) used in the second titration;

m is the mass, in grams, of the test portion (6.1);

0,023 51 is the mass, in grams, of sodium hexafluorosilicate corresponding to 1 ml of exactly 0,5 N sodium hydroxide solution $\left(\text{namely } \frac{Na_2SiF_6}{4\ 000} \times 0{,}5\right)$.

NOTE — If the concentrations of the standard volumetric solutions used are not exactly as stated in the list of reagents, appropriate corrections must be applied.

8 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.

ANNEX

ISO PUBLICATIONS RELATING TO SODIUM HEXAFLUOROSILICATE FOR INDUSTRIAL USE

ISO 4281 — Determination of free acidity and total hexafluorosilicate content.

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

ISO 5441 — Determination of calcium content — EDTA titrimetric method.

ISO 5442 — Determination of sulphur compounds content — Iodometric method after reduction.

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

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