International Standard 5442

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Sodium hexafluorosilicate for industrial use — Determination of sulphur compounds content — Iodometric method after reduction

Hexafluorosilicate de sodium à usage industriel — Dosage des composés soufrés — Méthode par réduction et iodométrie

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Foreword

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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 5442 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	Israel	South Africa, Rep. of
Brazil	Italy	Spain
Chile	Korea, Rep. of	Switzerland
Czechoslovakia	Mexico	Thailand
France	Netherlands	Turkey
Germany, F.R.	Philippines	United Kingdom
Hungary	Poland	Yugoslavia
India	Romania	

No member body expressed disapproval of the document.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC)

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Sodium hexafluorosilicate for industrial use — Determination of sulphur compounds content — Iodometric method after reduction

WARNING — Sodium hexafluorosilicate is poisonous if taken internally. Breathing of the dust should be avoided. Contact with skin and eyes 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 an iodometric method, after reduction, for the determination of the sulphur compounds content of sodium hexafluorosilicate for industrial use.

The method is applicable to products having sulphur compounds contents, expressed as SO_4^{2-}, between 0,03 and 0,5 % (*m/m*).

2 References

ISO 383, *Laboratory glassware — Interchangeable conical ground joints.*

ISO 5444, *Sodium hexafluorosilicate for industrial use — Determination of loss in mass at* 105 °C.

3 Principle

Reduction of the sulphur compounds in a test portion to hydrogen sulphide by heating with a mixture of hydriodic and phosphinic (hypophosphorous) acids, in the presence of hydrochloric acid.

Entrainment of the hydrogen sulphide, in a current of nitrogen and absorption in an excess of ammoniacal cadmium acetate solution. Liberation of the absorbed hydrogen sulphide in the presence of an excess of standard volumetric iodate/iodide solution.

Back-titration of the excess iodine with standard volumetric sodium thiosulphate solution using starch as indicator.

NOTE — The method is intended for the determination of the sulphate content, but any sulphides, and other compounds which will be reduced to sulphide by the reduction procedure specified, will be included in the result.

4 Reagents

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

4.1 Nitrogen, oxygen free, of minimum purity 99,8 %, contained in a cylinder fitted with a suitable pressure-reducing valve.

4.2 Hydrochloric acid, 145 g/l solution.

4.3 Reducing solution.

Place in a 1000 ml flask fitted with three ground necks and a bulb reflux condenser, in the following order and under a current of the nitrogen (4.1),

— 100 ml of hydriodic acid solution, ϱ approximately 1,71 g/ml about 57 % (*m/m*) solution;

— 25 ml of phosphinic (hypophosphorous) acid (H_3PO_2) solution, ϱ approximately 1,21 g/ml, about 50 % (*m/m*) solution;

— 100 ml of hydrochloric acid solution, ϱ approximately 1,19 g/ml, about 38 % (*m/m*) solution.

Fit the reflux condenser to the flask and, while bubbling a gentle current of the nitrogen (4.1) through the mixture, boil under reflux for about 4 h.

Then cool to ambient temperature, maintaining the current of nitrogen.

Store the reagent away from direct sunlight in a dark-glass flask, fitted with a ground glass stopper, and in a nitrogen atmosphere obtained by purging the flask initially with the nitrogen (4.1).

The solution is stable for several weeks.

NOTE — This reagent should be prepared in a fume cupboard so as to remove liberated hydrogen chloride.

4.4 Cadmium acetate, ammoniacal solution.

Place 16,4 g of cadmium acetate dihydrate [$(CH_3COO)_2Cd.2H_2O$] in a 1 000 ml one-mark volumetric flask and dissolve in 100 to 200 ml of water. Add 600 ml of ammonium hydroxide solution, ϱ approximately 0,88 g/ml, dilute with water to the mark and mix.

4.5 Potassium iodate/iodide, standard reference solution, $c(1/6\ KIO_3)$ = 0,1 mol/l.

Dry the potassium iodate in a electric oven at 100 °C for 2 h and cool in a desiccator. Weigh, to the nearest 0,000 2 g,

3,567 g of the dried material and place in a beaker of suitable capacity. Add approximately 40 g of iodate-free potassium iodide and 5 ml of a 40 g/l sodium hydroxide solution. Dissolve in water, transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

4.6 Sodium thiosulphate, standard volumetric solution, $c(Na_2S_2O_3.5H_2O) = 0,1$ mol/l.

4.6.1 Preparation of the solution

Place 25 g of sodium thiosulphate pentahydrate ($Na_2S_2O_3.5H_2O$) in a beaker of suitable capacity and dissolve in water. Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, add 1 ml of chloroform, dilute to the mark and mix.

4.6.2 Standardization of the solution

Place 10,0 ml of the potassium iodate/iodide standard reference solution (4.5) in a 250 ml ground glass-stoppered conical flask containing 100 ml of water. Add 40 ml of the hydrochloric acid solution (4.2). Stopper immediately and mix. Using the burette (5.2), titrate with the sodium thiosulphate solution (4.6.1) to a pale straw colour. Add approximately 1 ml of the starch solution (4.7) and continue the titration until the blue colour is discharged.

The exact concentration c, in moles of $Na_2S_2O_3.5H_2O$ per litre, of the sodium thiosulphate solution is given by the formula

$$\frac{0,1 \times 10}{V} = \frac{1}{V}$$

where

0,1 is the concentration, in moles of $(1/6\ KIO_3)$ per litre, of the potassium iodate/iodide standard reference solution (4.5);

V is the volume, in millilitres, of the solution used for the titration;

10 is the volume, in millilitres, of the potassium iodate/iodide standard reference solution (4.5) taken.

4.7 Starch, 10 g/l solution, freshly prepared.

Triturate 1,0 g of soluble starch with 5 ml of water and, while stirring, pour the mixture into 100 ml of boiling water; if necessary, boil for a few minutes, then cool.

Discard the solution after 2 weeks.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Apparatus for reduction, entrainment and absorption.

A suitable apparatus (see the figure), fitted with ground glass joints complying with ISO 383, consists of :

5.1.1 Flask, three-necked, pear-shaped, of capacity 25 ml.

5.1.2 Liebig condenser.

5.1.3 Nitrogen inlet tube.

5.1.4 Glass connecting tube.

5.1.5 Conical flask, of capacity 250 ml, fitted with a ground glass stopper.

5.1.6 Heat screen, of suitable material.

5.1.7 Water bath.

5.2 Burette, of capacity 10 ml, graduated in 0,02 ml.

6 Test sample

Use the residue from the determination of the loss in mass at 105 °C (see ISO 5444) as the test sample.

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 6) into the clean dry flask (5.1.1) of the apparatus.

7.2 Blank test

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

7.3 Determination

Assemble the apparatus (5.1), placing the flask (5.1.1) in the water bath (5.1.7).

Pour 100 ml of water and 10 ml of the ammoniacal cadmium acetate solution (4.4) into the conical flask (5.1.5) and place it in position to receive the hydrogen sulphide from the flask (see the figure).

Bring the water bath (5.1.7) to the boil, add 10 ml of the reducing solution (4.3) to the flask (5.1.1) and pass a stream of the nitrogen (4.1) through to sweep the hydrogen sulphide into the ammoniacal cadmium acetate solution. The rate shall be such that an almost continuous stream of bubbles is observed in the conical flask (5.1.5). Heat for 15 min after the hydrogen sulphide begins to be absorbed. This is shown by the solution turning yellow.

Dismantle the apparatus and wash the connecting tube (5.1.4) with a few millilitres of water collecting the washings in the conical flask (5.1.5).

Place 10,0 ml of the standard reference potassium iodate/iodide solution (4.5) in the conical flask (5.1.5). Add quickly 40 ml of the hydrochloric acid solution (5.2). Stopper the flask immediately and shake for 1 min. Unstopper the flask and dip the end of the connecting tube (5.1.4) into the solution so as to dissolve any adhering cadmium sulphide. Rinse the tube with a little water, collecting the washings in the flask, stopper immediately and shake the flask for 1 min.

Titrate the excess of the iodine with the standard volumetric sodium thiosulphate solution (4.6), using the burette (5.2).

8 Expression of results

The sulphur compounds content, expressed as a percentage by mass of SO_4^{2-}, is given by the formula

$$\frac{(0{,}1\ V_1 - cV_2) - (0{,}1\ V_1 - cV_0)}{m} \times 0{,}004\ 8 \times 100$$

$$= \frac{c(V_0 - V_2)}{m} \times 0{,}48$$

where

V_0 is the volume, in millilitres, of the standard volumetric sodium thiosulphate solution (4.6) used for the blank test;

V_1 is the volume, in millilitres, of the standard reference potassium iodate/iodide solution (4.5) added to the conical flask (5.1.5);

V_2 is the volume, in millilitres, of the standard volumetric sodium thiosulphate solution (4.6) used for the determination;

c is the exact concentration, in moles of $Na_2S_2O_3.5H_2O$ per litre, of the standard volumetric sodium thiosulphate solution (4.6) determined as specified in 4.6.2;

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

0,004 8 is the mass, in grams, of SO_4^{2-} corresponding to 1,00 ml of sodium thiosulphate solution, $c(Na_2S_2O_3.5H_2O) = 0{,}100$ mol/l.

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.

10 Bibliography

[1] LUKE, C.L., *Ind. Eng. Chem. Anal.* Ed. 15, 1943.

[2] ARCHER, E.E., *Analyst*, **81**, 1956.

Annex

ISO publications relating to sodium hexafluorosilicate for industrial use

(This annex does not form part of the Standard.)

ISO 4281 — Free acidity and sodium 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.

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

ISO 6229 — Determination of free silica content — Gravimetric method.