
International Standard



5442

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

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

First edition — 1980-10-15

UDC 553.634 : 546.22 : 543.242.3

Ref. No. ISO 5442-1980 (E)

Descriptors : chemical compounds, sodium fluorosilicates, chemical analysis, determination of content, sulphates, iodometric analysis, reduction analysis.

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)

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 $[(\text{CH}_3\text{COO})_2\text{Cd}\cdot 2\text{H}_2\text{O}]$ 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, $c(1/6 \text{ KIO}_3) = 0,1 \text{ mol/l}$.

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,