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International Standard 6229

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Sodium hexafluorosilicate for industrial use — Determination of free silica content — Gravimetric method

Hexafluorosilicate de sodium à usage industriel — Dosage de la silice libre — Méthode gravimétrique

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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 6229 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in October 1978.

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

Australia	Germany, F.R.	Philippines
Austria	Hungary	Poland
Belgium	India	Romania
Bulgaria	Israel	South Africa, Rep. of
China	Italy	Switzerland
Czechoslovakia	Korea, Rep. of	United Kingdom
Egypt, Arab Rep. of	Mexico	USSR
France	Netherlands	Yugoslavia

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).

Printed in Switzerland

Sodium hexafluorosilicate for industrial use — Determination of free silica content — Gravimetric method

WARNING — Sodium hexafluorosilicate is poisonous if taken internally. Breathing of the dust should be avoided. Contact with eyes and skin shall 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 gravimetric method for the determination of the free silica content of sodium hexafluorosilicate for industrial use.

The method is applicable to products having free silica contents, expressed as SiO_2, equal to or greater than 0,05 % (*m/m*).

2 REFERENCE

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

3 PRINCIPLE

Extraction from a test portion of compounds soluble in acetic acid solution and weighing of the residue after heating at 600 °C. Removal from the residue, of silica by evaporation with hydrofluoric acid solution and of residual fluorides by heating with ammonia solution, and reweighing of the residue after heating at 600 °C.

4 REAGENTS

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

4.1 Hydrofluoric acid, ρ approximately 1,14 g/ml, about 40 % (*m/m*) solution.

4.2 Acetic acid, glacial, ρ approximately 1,05 g/ml.

4.3 Ammonia solution, ρ approximately 0,88 g/ml, about 34 % (*m/m*) solution.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Platinum crucible, capacity 35 to 50 ml.

5.2 Electric furnace, capable of being controlled at 600 ± 10 °C.

6 SAMPLING[1)]

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

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0,000 1 g, about 1 g of the test sample, finely ground and previously dried at 105 °C (see clause 6).

7.2 Determination

Quantitatively transfer the test portion (7.1) to a 400 ml beaker and add 5 ml of the acetic acid (4.2) and 200 ml of water. Bring the contents of the beaker to the boil and boil gently for 10 min with continuous stirring. Allow to stand, maintaining the temperature for 10 min. Filter first the decanted supernatant liquid and then the suspended residue, ensuring that the residue is transferred quantitatively on to the filter paper used for this purpose.[2)] Wash the residue four or five times with the hot water that was used to rinse the beaker. Transfer the filter paper and residue to the clean, dry platinum crucible (5.1), previously weighed to the nearest 0,000 1 g. Heat the crucible and contents for 45 min in the furnace (5.2), controlled at 600 ± 10 °C, transfer to a desiccator, allow to cool, and weigh to the nearest 0,000 1 g. Repeat the operations of heating (for 30 min), cooling and weighing until the difference between two successive weighings does not exceed 0,000 2 g.

Then add 2 ml of the hydrofluoric acid solution (4.1) to the crucible (5.1) and carefully evaporate the contents of

1) An International Standard concerning sampling of solid chemical products is in preparation.

2) Whatman, Grade N° 44, Ashless or an equivalent grade, is suitable for this purpose.

the crucible to dryness in a fume cupboard. Allow to cool, add a small filter paper of known ash to the crucible, moisten it with 2 drops of the ammonia solution (4.3) and heat gently to dryness. Heat the crucible and contents for 45 min in the furnace (5.2), controlled at 600 ± 10 °C, transfer to a desiccator, allow to cool, and weigh to the nearest 0,000 1 g. Repeat the operations of heating (for 30 min), cooling and weighing until the difference between two successive weighings does not exceed 0,000 2 g.

8 EXPRESSION OF RESULTS

The free silica content, expressed as a percentage by mass of SiO_2, is given by the formula

$$\frac{m_1 - (m_2 + m_3)}{m_0} \times 100$$

where

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

m_1 is the mass, in grams, of the residue before heating with hydrofluoric acid;

m_2 is the mass, in grams, of the residue after heating with hydrofluoric acid;

m_3 is the mass, in grams, of the filter paper ash.

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 – Determination of free acidity and total hexafluorosilicate content – Titrimetric method.

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.

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