INTERNATIONAL STANDARD 1690

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Sodium and potassium silicates for industrial use — Determination of silica content — Gravimetric method by insolubilization

Silicates de sodium et de potassium à usage industriel — Dosage de la silice — Méthode gravimétrique par insolubilisation

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FOREWORD

ISO (the International Organization for Standardization) is a worlwide 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.

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Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47 has reviewed ISO Recommendation R 1690 and found it technically suitable for transformation. International Standard ISO 1690 therefore replaces ISO Recommendation R 1690-1970 to which it is technically identical.

ISO Recommendation R 1690 was approved by the Member Bodies of the following countries :

Australia	Hungary	Portugal
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No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 19760 into an International Standard.

Printed in Switzerland

Sodium and potassium silicates for industrial use — Determination of silica content — Gravimetric method by insolubilization

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric method by insolubilization for the determination of the silica content of sodium and potassium silicates for industrial use.

2 REFERENCE

ISO 1686, *Sodium and potassium silicates for industrial use — Samples and methods of test — General.*

3 PRINCIPLE

Insolubilization of silica by evaporation to dryness of a test portion previously acidified with hydrochloric acid.

Dissolution of the soluble salts, filtration and washing of the insoluble matter. Second insolubilization by evaporation of the filtrate and washings under the same conditions. Further dissolution of the soluble salts, filtration and washing of the second insoluble matter. Calcination and weighing of the two lots of insoluble matter simultaneously.

Volatilization of silica by heating after addition of hydrofluoric and sulphuric acids and weighing of the residue after calcination.

The difference in mass represents the silica present in the test portion.

4 REAGENTS

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

4.1 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution or approximately 12 N.

4.2 Sulphuric acid, ρ approximately 1,84 g/ml, about 96 % (*m/m*) solution or approximately 36 N.

4.3 Hydrofluoric acid, ρ approximately 1,15 g/ml, about 40 % (*m/m*) solution.

4.4 Silver nitrate, 10 g/l nitric solution.

Dissolve 1 g of silver nitrate in water, add 10 ml of nitric acid solution, ρ approximately 1,40 g/ml, and make up the volume to 100 ml.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Platinum crucible of height approximately 40 mm and top diameter 30 mm, with lid.

5.2 Electric oven, capable of being controlled at 100 to 105 °C and at 110 to 120 °C.

5.3 Electric furnace, capable of being controlled at 950 to 975 °C.

6 PROCEDURE

6.1 Test portion and preparation of test solution

Weigh, to the nearest 0,01 g, 10 ± 1 g of the test sample (see ISO 1686) and place it in a 250 ml one-mark volumetric flask. Dissolve with water, dilute to the mark and mix.

6.2 Determination

Take 25,0 ml (corresponding to approximately 1 g of test sample) of the test solution (6.1) and place in a porcelain dish of suitable capacity. Carefully add, while stirring, 5 ml of the hydrochloric acid solution (4.1).

Cover the dish with a watch-glass supported by a glass triangle and place it on a boiling water bath and evaporate to dryness.

Heat the residue in the oven (5.2) controlled at 110 to 120 °C for at least 1 h.

After cooling, add 5 ml of the hydrochloric acid solution (4.1) and 10 ml of water.

Place on the boiling water bath for 5 to 10 min and stir to dissolve the soluble salts.

Filter through an ashless filter paper of medium porosity, and collect the filtrate quantitatively. Transfer the insoluble product quantitatively onto the filter by means of hot water. Wash with hot water, adding the rinsing water to the filtrate. Continue washing until 10 ml of the filtrate from the funnel remain clear 5 min after the addition of 10 ml of the nitric solution of silver nitrate (4.4). (Discard this portion of the filtrate.)

Transfer the filtrate and the rinsing water quantitatively into the dish already used and evaporate to dryness on a boiling water bath. Heat the residue in the oven (4.2) at 110 to 120 °C for at least 1 h. After cooling, add 5 ml of the hydrochloric acid solution (4.1) and 10 ml of water.

Place on the boiling water bath for 5 to 10 min and stir in order to dissolve the soluble salts. Filter and wash the insoluble matter on another filter paper by following the procedure given above for the first filtration.

Fold the filter papers containing the insoluble matter and place them in the platinum crucible (5.1).

Dry in the oven controlled at 100 to 105 °C, then ignite the paper with a low flame until the combustion stops.

Allow to cool, and wet the residue with a few drops of the sulphuric acid solution (4.2).

Place the crucible and its contents on a hot-plate and evaporate the sulphuric acid. When the emission of white fumes has ceased, place the crucible in the furnace (5.3), controlled at 950 to 975 °C, for 15 min in order to calcine its contents. Allow to cool in a desiccator to ambient temperature and weigh to the nearest 0,000 2 g.

Wet the residue with a few drops of the sulphuric acid solution (4.2) and add, with care, 2 to 3 ml of the hydrofluoric acid solution (4.3).

Heat gently on a hot-plate in order to volatilize the hydrofluoric acid and heat more strongly in order to evaporate the sulphuric acid. If any residue remains in the crucible, repeat the treatment with the sulphuric and hydrofluoric acids.

Finally, calcine in the furnace controlled at 950 to 975 °C for 15 min, allow to cool in a desiccator to ambient temperature and weigh to the nearest 0,000 2 g.

7 EXPRESSION OF RESULTS

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

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

where

m_0 is the mass, in grams, of test portion contained in the volume of the solution used;

m_1 is the mass, in grams, of the crucible containing the insoluble product before the treatment with hydrofluoric acid;

m_2 is the mass, in grams, of the crucible after the treatment with hydrofluoric acid.

8 ACCURACY OF THE METHOD

The results obtained using this method are reproducible to the nearest ± 0,2 % (*m/m*), in absolute value.

9 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 operations not included in this International Standard or the International Standard to which reference is made, or regarded as optional.

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