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R 1690

SODIUM AND POTASSIUM SILICATES FOR INDUSTRIAL USE

DETERMINATION OF SILICA CONTENT

GRAVIMETRIC METHOD BY INSOLUBILIZATION

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BRIEF HISTORY

The ISO Recommendation R 1690, *Sodium and potassium silicates for industrial use – Determination of silica content – Gravimetric method by insolubilization*, was drawn up by Technical Committee ISO/TC 47, *Chemistry*, the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1690 which was circulated to all the ISO Member Bodies for enquiry in December 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia
Austria
Belgium
Brazil
Colombia
Czechoslovakia
France
Germany
Greece
Hungary
India
Iran
Israel
Italy
Japan
Netherlands
New Zealand
Peru
Poland
Portugal
Romania
South Africa, Rep. of
Spain
Switzerland
Thailand
Turkey
U.A.R.
United Kingdom
U.S.S.R.
Yugoslavia

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided to accept it as an ISO RECOMMENDATION.

ISO Recommendation
R 1690
July 1970

SODIUM AND POTASSIUM SILICATES

FOR INDUSTRIAL USE

DETERMINATION OF SILICA CONTENT

GRAVIMETRIC METHOD BY INSOLUBILIZATION

1. SCOPE

This ISO Recommendation describes a gravimetric method for the determination of silica content in sodium and potassium silicates for industrial use.

2. PRINCIPLE

Insolubilization of silica by evaporation to dryness of the 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 SiO_2 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.

3. REAGENTS

Only distilled water or water of equal purity should be used in the test.

3.1 *Hydrochloric acid*, approximately ρ 1.19 (g/ml), 38 % (m/m) or 12 N solution.

3.2 *Sulphuric acid*, approximately ρ 1.84 (g/ml), 96 % (m/m) or 36 N solution.

3.3 *Hydrofluoric acid*, approximately ρ 1.15 (g/ml), 40 % (m/m) solution.

3.4 *Silver nitrate*, 10 g/l solution, acidified with nitric acid.
Dissolve 1 g of silver nitrate in water, add 10 ml of nitric acid solution, approximately ρ 1.4 (g/ml) and make up the volume to 100 ml.

4. APPARATUS

Ordinary laboratory apparatus and

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

4.2 *Oven* controlled at 110 to 120 °C.

4.3 *Muffle furnace* controlled at 950 °C.

5. PROCEDURE

5.1 Test portion

Weigh, to the nearest 0.01 g, 10 ± 1 g of the test sample and introduce it into a 250 ml one-mark volumetric flask. Dissolve with water, dilute to the mark and mix thoroughly. Take a test portion of 25.0 ml (corresponding to approximately 1 g of the test sample) from this solution.

5.2 Determination

Place the test portion (5.1) in a porcelain dish of suitable capacity. Carefully add, while stirring, 5 ml of the hydrochloric acid solution (3.1).

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 (4.2) at 110 to 120 °C for at least 1 hour.

After cooling, add 5 ml of the hydrochloric acid solution (3.1) and 10 ml of water.

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

Filter through an ashless paper filter of medium porosity, and collect the filtrate. 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 minutes after the addition of 10 ml of the silver nitrate solution (3.4). (Discard this test solution.)

Transfer the filtrate and the rinsing water 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 hour. After cooling, add 5 ml of the hydrochloric acid solution (3.1) and 10 ml of water.

Place on the boiling water bath for 5 to 10 minutes 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 (4.1).

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

Cool, and wet the residue with a few drops of the sulphuric acid solution (3.2).

Place the crucible and its contents on a hotplate and evaporate the sulphuric acid. When the emission of white fumes has ceased, place the crucible in the furnace (4.3), at approximately 950 °C for 15 minutes in order to calcine its contents.

Cool in a desiccator to ambient temperature and weigh to the nearest 0.2 mg.

Wet the residue with a few drops of the sulphuric acid solution (3.2) and add, with care, 2 to 3 ml of the hydrofluoric acid solution (3.3).

Heat gently on a hotplate 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 (4.3) at approximately 950 °C for 15 minutes, cool in a desiccator to ambient temperature and weigh to the nearest 0.2 mg.