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ISO RECOMMENDATION

R 984

SODIUM HYDROXIDE FOR INDUSTRIAL USE

DETERMINATION OF SILICA CONTENT

GRAVIMETRIC METHOD BY INSOLUBILIZATION

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BRIEF HISTORY

The ISO Recommendation R 984, *Sodium hydroxide 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, in 1966, to the adoption of a Draft ISO Recommendation.

In December 1966, this Draft ISO Recommendation (No. 1093) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria	Ireland	Spain
Belgium	Israel	Switzerland
Chile	Japan	Thailand
Cuba	Korea, Dem. P. Rep. of	Turkey
Czechoslovakia	Netherlands	U.A.R.
France	New Zealand	United Kingdom
Germany	Poland	U.S.S.R.
Hungary	Portugal	Yugoslavia
India	Romania	
Iran	South Africa, Rep. of	

Two Member Bodies opposed the approval of the Draft :

Italy
U.S.A.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in February 1969, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 984 February 1969

SODIUM HYDROXIDE FOR INDUSTRIAL USE

DETERMINATION OF SILICA CONTENT

GRAVIMETRIC METHOD BY INSOLUBILIZATION

1. SCOPE

This ISO Recommendation describes a gravimetric method by insolubilization for the determination of silica content in sodium hydroxide for industrial use.

2. FIELD OF APPLICATION

The method is applicable to the determination of silica (SiO_2) content greater than or equal to 0.01 % (m/m) calculated on NaOH.

3. PRINCIPLE

Insolubilization of silica by evaporation to dryness of the test portion, previously dissolved and acidified (by means of hydrochloric acid).

Solution of the soluble salts and filtration of the insoluble matter containing silica. Washing, recovery of silica contained in the filtrate and washings, filtration, washing, drying, ignition and weighing of the insoluble matter.

After treatment with hot hydrofluoric and sulphuric acids to remove the silica, further ignition and weighing.

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

4. REAGENTS

Distilled water or water of equivalent purity should be used in the test.

4.1 *Hydrochloric acid*, approximately, $d = 1.19$, 38 % (m/m) or 12 N solution.

4.2 *Hydrofluoric acid*, approximately, $d = 1.15$, 40 % (m/m) solution.

4.3 *Sulphuric acid*, approximately, $d = 1.18$, 25 % (m/m) or 6 N solution.

4.4 *Silver nitrate*, 5 g/l nitric solution.
Dissolve 0.5 g of silver nitrate in a small amount of water, add 10 ml of approximately $d = 1.4$ nitric acid solution and dilute to 100 ml.

4.5 *Methyl orange*, 0.5 g/l solution.
Dissolve 0.05 g of methyl orange in water and dilute to 100 ml.

5. APPARATUS

5.1 *Ordinary laboratory apparatus.*

5.2 *Platinum crucible*, approximately 30 mm in top diameter and 30 mm deep.

6. PROCEDURE

6.1 Test portion

In a weighing bottle of approximately 200 ml capacity, fitted with a ground glass stopper, weigh to the nearest 0.1 g a mass of the test sample (solid or liquid)* containing between 40 and 45 g of NaOH.

6.2 Determination

6.2.1 Transfer the test portion (6.1) quantitatively to a flat-bottomed porcelain dish with a diameter of approximately 15 cm. If the product is solid, dissolve in about 200 ml of water; if the product is liquid, dilute to approximately 200 ml.

6.2.2 Add 2 drops of the methyl orange solution (4.5), neutralize with the hydrochloric acid solution (4.1), added slowly, and then add a 5 ml excess of the acid. Evaporate to dryness on a boiling water bath, loosen the residue and crush it by means of a flat-tipped stirrer, then place in an oven at 130 to 140 °C for at least 2 hours.

6.2.3 Allow to cool to room temperature. Moisten the residue with about 10 ml of the hydrochloric acid solution (4.1), taking care that the whole mass is impregnated, and after a few minutes add 200 ml of boiling water.

6.2.4 Place on a boiling water bath for approximately 15 minutes and stir to dissolve the soluble salts completely.

6.2.5 Filter on an ashless, slow-speed filter paper of about 110 mm diameter (pore diameter between 0.4 and 1 μm). Wash the insoluble matter and filter paper with boiling water until 10 ml of the liquid flowing from the funnel remain clear 5 minutes after the addition of 10 ml of the nitric solution of silver nitrate (4.4). (Discard this solution.)

6.2.6 Transfer the filtrate and washings to the dish used previously, and evaporate to dryness on a boiling water bath. Place in the oven at 130 to 140 °C for at least 1 hour. After cooling, add 10 ml of the hydrochloric acid solution (4.1) and 20 ml of water. Place on the boiling water bath for 5 to 10 minutes and mix in order to dissolve the soluble salts. Filter and wash the residue on an ashless, slow-speed filter paper following the same procedure as indicated for the first filtration (see clause 6.2.5).

6.2.7 Place the two filter papers and their contents in the platinum crucible (5.2). Dry in an oven at 110 °C, then ignite in a furnace, gently at first to char the filter paper, then at 1100 to 1200 °C for 1 hour. Cool in a desiccator to room temperature and weigh.

6.2.8 Moisten the residue with 3 or 4 drops of the sulphuric acid solution (4.3) and add approximately 15 ml of the hydrofluoric acid solution (4.2). Slowly evaporate to dryness on a hotplate, ignite in a furnace at 1100 to 1200 °C, cool in a desiccator to room temperature and re-weigh.

* See ISO Recommendation R 977, *Sodium hydroxide for industrial use – Preparation and storage of test sample*, clause 2.2.