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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 985

SODIUM HYDROXIDE FOR INDUSTRIAL USE

DETERMINATION OF SILICA CONTENT

GRAVIMETRIC METHOD BY PRECIPITATION

OF THE QUINOLINE-SILICOMOLYBDIC COMPLEX

1st EDITION
February 1969

For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 985, *Sodium hydroxide for industrial use – Determination of silica content – Gravimetric method by precipitation of the quinoline-silicomolybdic complex,* 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. 1094) 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	Italy	Thailand
Cuba	Japan	Turkey
Czechoslovakia	Netherlands	U.A.R.
France	New Zealand	United Kingdom
Germany	Poland	U.S.A.
Hungary	Portugal	U.S.S.R.
India	Romania	
Iran	South Africa, Rep. of	

No Member Body opposed the approval of the Draft.

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 985 February 1969

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DETERMINATION OF SILICA CONTENT

GRAVIMETRIC METHOD BY PRECIPITATION

OF THE QUINOLINE-SILICOMOLYBDIC COMPLEX

1. SCOPE

This ISO Recommendation describes a gravimetric method for the determination of the silica content in sodium hydroxide for industrial use by precipitation of the quinoline-silicomolybdic complex.

2. FIELD OF APPLICATION

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

3. PRINCIPLE

Dissolution of a test portion and acidification by hydrochloric acid solution, formation of the silicomolybdic complex and precipitation of a high molecular weight compound by quinoline, filtration, washing, drying at 150 °C and weighing of the compound.

4. REACTION

The basic reaction (precipitation by quinoline introduced in the form of hydrochloride) is as follows :

$$SiO_2.12MoO_3.2H_2O + 4C_9H_7N.HCl = (C_9H_7N)_4.SiO_2.12MoO_3.2H_2O + 4HCl$$

5. REAGENTS

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

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

5.2 *Ammonium molybdate,* 100 g/l solution.
Dissolve 10 g of ammonium molybdate tetrahydrate $[(NH_4)_6Mo_7O_{24}.4H_2O]$ in water and dilute to 100 ml.

5.3 *Oxalic acid,* 100 g/l solution.
Dissolve 10 g of oxalic acid in water and dilute to 100 ml.

5.4 *Quinoline*, 20 g/l hydrochloric solution.
Dissolve 20 ml of quinoline, approximately $d = 1.093$ to 1.096, in 25 ml of the hydrochloric acid solution (5.1). Stir and dilute to 1000 ml.

5.5 *Washing solution.*
Dilute 25 ml of the quinoline hydrochloric solution (5.4) to 1000 ml.

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

6. APPARATUS

6.1 *Ordinary laboratory apparatus.*

6.2 *Filter crucible* with sintered disk of porosity between 5 and 15 μm.

7. PROCEDURE

7.1 Test portion

In a weighing bottle of approximately 100 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 20 ± 0.1 g of NaOH (see clause 9.1).

7.2 Blank test

Together with the analysis and following the same procedure, carry out a blank test using the same quantities of all reagents (see clause 9.2).

7.3 Determination

Place the test portion (7.1) in a beaker of suitable capacity (600 ml, for example). In the case of solid material, dissolve the test portion in about 100 ml of water; in the case of liquid material, dilute to approximately 100 ml.

Add 2 drops of the methyl orange solution (5.6), neutralize by slowly adding the hydrochloric acid solution (5.1) and add an excess of approximately 3 ml of the acid.

Cool to room temperature, transfer to a 250 ml one-mark volumetric flask, dilute to the mark, and mix thoroughly.

Quantitatively transfer the solution back to the 600 ml beaker using only a minimum volume of rinsing water. Add 25 ml of the ammonium molybdate solution (5.2) and wait 10 minutes so as to allow the silicomolybdic complex to form.

Then add 25 ml of the hydrochloric acid solution (5.1) and 20 ml of the oxalic acid solution (5.3). Stir for 30 seconds to promote the decomposition of any phosphomolybdic complex that may have formed, then, while still stirring, add 25 ml of the quinoline solution (5.4).

Heat to approximately 80 °C, stirring from time to time so as to obtain a precipitate which can easily be filtered, and then cool to room temperature.

Weigh the filter crucible (6.2), previously dried in an oven at 150 °C and cooled to room temperature in a desiccator. Filter the decanted solution through the filter crucible, maintaining a reduced pressure by means of a filter pump or a vacuum pump.

Wash the precipitate once by decantation in the beaker with the washing solution (5.5), transfer the precipitate to the filter crucible and wash six times.

Drain by keeping under vacuum for 1 minute and dry the filter crucible with its contents in an oven at 150 °C for 1 hour. Remove the filter crucible from the oven, cool to room temperature in a desiccator and quickly weigh.

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