47

INTERNATIONAL STANDARD 985

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Sodium hydroxide for industrial use — Determination of silica content — Gravimetric method by precipitation of quinoline molybdosilicate

Hydroxyde de sodium à usage industriel — Dosage de la silice — Méthode gravimétrique par précipitation du molybdosilicate de quinoléine

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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.

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 985 and found it technically suitable for transformation. International Standard ISO 985 therefore replaces ISO Recommendation R 985-1969 to which it is technically identical.

ISO Recommendation R 985 was approved by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the Recommendation.

The Member Body of the following country disapproved the transformation of ISO/R 985 into an International Standard :

United Kingdom

Printed in Switzerland

Sodium hydroxide for industrial use — Determination of silica content — Gravimetric method by precipitation of quinoline molybdosilicate

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric method for the determination of the silica content of sodium hydroxide for industrial use, by precipitation of quinoline molybdosilicate.

The method is applicable to products having a silica (SiO_2) content, calculated on NaOH, equal to or greater than 0,001 % (*m/m*).

2 REFERENCE

ISO 3195, *Sodium hydroxide for industrial use — Sampling — Test sample — Preparation of the main solution for carrying out certain determinations.*

3 PRINCIPLE

Dissolution of a test portion and acidification by hydrochloric acid solution, formation of the molybdosilicate and precipitation of a high molecular weight compound by quinoline.

Filtration, washing, drying at 150 ± 2 °C and weighing of the compound.

4 REACTION

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

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

5 REAGENTS

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

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

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.

5.4 Quinoline, 20 g/l hydrochloric solution.

Dissolve 20 g of quinoline, ρ approximately 1,093 to 1,096 g/ml, in 25 ml of the hydrochloric acid solution (5.1). Stir and dilute to 1 000 ml.

5.5 Washing solution.

Dilute 25 ml of the quinoline hydrochloric solution (5.4) to 1 000 ml.

5.6 Methyl orange, 0,5 g/l solution.

6 APPARATUS

Ordinary laboratory apparatus and

6.1 Filter crucible, with sintered disk of porosity grade P 16 (pore diameter between 10 and 16 μm).

6.2 Electric oven, capable of being controlled at 150 ± 2 °C.

7 PROCEDURE

7.1 Test portion

In a weighing bottle of capacity approximately 100 ml, fitted with a ground glass stopper, weigh, to the nearest 0,1 g, a mass of the solid or liquid test sample corresponding to 20 ± 0,1 g of NaOH (see ISO 3195).

NOTE — If the SiO_2 content of the test portion is higher than 0,010 g, it will be advisable to repeat the determination using a smaller test portion.

7.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure, and using the same quantities of all reagents as used for the determination.

NOTE — The mass of the precipitate weighed shall not exceed 0,005 g.

7.3 Determination

Place the test portion (7.1) in a beaker of suitable capacity (for example 600 ml). 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.

Allow to cool to ambient temperature, transfer quantitatively to a 250 ml one-mark volumetric flask, dilute to the mark, and mix.

Transfer the solution quantitatively 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 min to allow the molybdosilicate 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 s to promote the decomposition of any phosphomolybdate 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 allow to cool to ambient temperature.

Weigh, to the nearest 0,000 1 g, the filter crucible (6.1) previously dried in the oven (6.2) controlled at 150 ± 2 °C and allowed to cool to ambient 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 min and dry the filter crucible with its contents in the oven controlled at 150 ± 2 °C for 1 h. Remove the filter crucible from the oven, allow to cool to ambient temperature in a desiccator and quickly weigh to the nearest 0,000 1 g.

8 EXPRESSION OF RESULTS

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

$$(m_1 - m_2) \times \frac{1}{38,94} \times \frac{100}{m_0} = 2,568 \frac{(m_1 - m_2)}{m_0}$$

where

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

m_1 is the mass, in grams, of quinoline molybdosilicate obtained from the test portion;

m_2 is the mass, in grams, of quinoline molybdosilicate obtained from the blank test;

$\frac{1}{38,94}$ is the conversion factor for $[(C_9H_7N)_4.12MoO_3.SiO_2.2H_2O]$ to SiO_2.

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

ANNEX

ISO PUBLICATIONS RELATING TO SODIUM HYDROXIDE FOR INDUSTRIAL USE

ISO 979 – Method of assay.

ISO 980 – Determination of carbonates content – Gas-volumetric method.

ISO 981 – Determination of chloride content – Mercurimetric method.

ISO 982 – Determination of sulphate content – Barium sulphate gravimetric method.

ISO 983 – Determination of iron content – 1,10-Phenanthroline photometric method.

ISO 984 – Determination of silica content – Reduced silicomolybdic complex photometric method.

ISO 985 – Determination of silica content – Gravimetric method by precipitation of quinoline molybdosilicate.

ISO 986 – Determination of calcium – EDTA complexometric method.

ISO 3195 – Sampling – Test sample – Preparation of the main solution for carrying out certain determinations.

ISO 3196 – Determination of carbon dioxide content – Titrimetric method.

ISO 3197 – Determination of chloride content – Photometric method.

ISO 3198 – Determination of sulphur compounds – Method by reduction and titrimetry.

ISO 3697 – Determination of calcium and magnesium contents – Flame atomic absorption spectrophotometric method.