INTERNATIONAL STANDARD



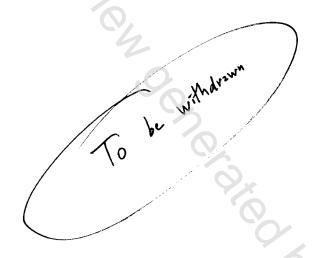
985

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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Descriptors: sodium hydroxide, chemical analysis, determination of content, silicon dioxide, gravimetric analysis.

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
Belgium Ireland
Chile Israel
Cuba Italy
Czechoslovakia Japan
Egypt, Arab Rep. of Netherl
France New Ze

Germany

Hungary India Italy
Japan
Netherlands
New Zealand
Poland
Portugal
Romania

South Africa, Rep. of

Spain Switzerland Thailand Turkey

United Kingdom U.S.A.

U.S.S.R. Yugoslavia

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

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:

$$12\text{MoO}_3.\text{SiO}_2.2\text{H}_2\text{O} + 4\text{C}_9\text{H}_7\text{N.HCI} \longrightarrow$$

$$(\text{C}_9\text{H}_7\text{N})_4.12\text{MoO}_3.\text{SiO}_2.2\text{H}_2\text{O} + 4\text{HCI}$$

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 μ m).
- **6.2** Electric oven, capable of being controlled at 150 \pm 2 $^{\circ}$ 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 $20 \pm 0,1$ 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 $0.005\,\mathrm{g}$.

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