INTERNATIONAL STANDARD 996

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

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

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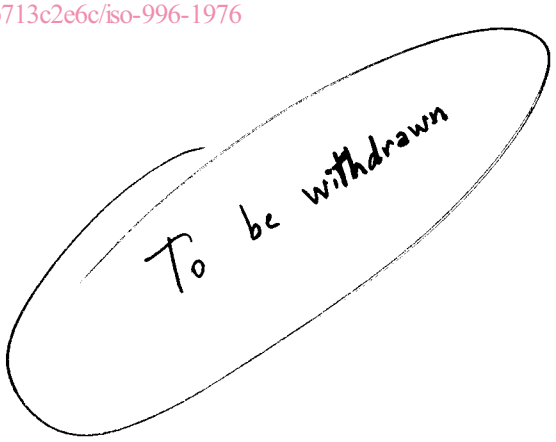

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FOREWORD

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 996 and found it technically suitable for transformation. International Standard ISO 996 therefore replaces ISO Recommendation R 996-1969 to which it is technically identical.

ISO Recommendation R 996 was approved by the Member Bodies of the following countries :

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

The Member Bodies of the following countries expressed disapproval of the Recommendation on technical grounds :

France*
Italy

* Subsequently, this Member Body approved the Recommendation.

The Member Body of the following country disapproved the transformation of ISO/R 996 into an International Standard :

United Kingdom

Printed in Switzerland

Potassium 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 potassium hydroxide for industrial use, by precipitation of quinoline molybdosilicate.

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

2 REFERENCE

ISO 2466, *Potassium 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 recognized analytical grade and only distilled water or water of equivalent purity.

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

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 KOH (see ISO 2466).

NOTE — If the test portion contains more than 0,010 g of SiO_2, recommence the determination using a smaller test portion.

7.2 Blank test

In parallel with the determination and following the same procedure, carry out a blank test using the same quantities of all reagents as used for the determination.

NOTE — The mass of 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 slowly 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.

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 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 (6.2) 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 of $[(C_9H_7N)_4.12MoO_3.SiO_2.2H_2O]$ 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 in the International Standard to which reference is made, or regarded as optional.

ANNEX

ISO PUBLICATIONS RELATING TO POTASSIUM HYDROXIDE FOR INDUSTRIAL USE

ISO 990 – Method of assay.

ISO 991 – Determination of carbonate content – Gas-volumetric method.

ISO 992 – Determination of chloride content – Mercurimetric method.

ISO 993 – Determination of sulphate content – Barium sulphate gravimetric method.

ISO 994 – Determination of iron content – 1,10-Phenanthroline photometric method.

ISO 995 – Determination of silica content – Reduced silicomolybdic complex photometric method.

ISO 996 – Determination of silica content – Gravimetric method by precipitation of quinoline molybdosilicate.

ISO 997 – Determination of calcium – EDTA (*di*sodium salt) complexometric method.

ISO 1550 – Determination of sodium content – Flame emission spectrophotometric method.

ISO 2466 – Sampling – Test sample – Preparation of the main solution for carrying out certain determinations.

ISO 2900 – Determination of carbon dioxide content – Titrimetric method.

ISO 3177 – Determination of chloride content – Photometric method.

ISO 3194 – Determination of sulphur compounds – Method by reduction and titrimetry.

ISO 3698 – Determination of calcium and magnesium contents – Flame atomic absorption method.