



Potassium hydroxide for industrial use – Determination of sulphate content – Barium sulphate gravimetric method

Hydroxyde de potassium à usage industriel – Dosage des sulfates – Méthode gravimétrique à l'état de sulfate de baryum

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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 993 and found it technically suitable for transformation. International Standard ISO 993 therefore replaces ISO Recommendation R 993-1969 to which it is technically identical.

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ISO Recommendation R 993 was approved by the Member Bodies of the following countries :

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

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

France*
Italy
U.S.A.

* Subsequently, this Member Body approved the Recommendation.

The Member Body of the following country disapproved the transformation of ISO/R 993 into an International Standard :

United Kingdom

Potassium hydroxide for industrial use – Determination of sulphate content – Barium sulphate gravimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a barium sulphate gravimetric method for the determination of the sulphate content of potassium hydroxide for industrial use.

The method is applicable to products having a sulphate content, expressed as potassium sulphate and calculated on KOH, equal to or greater than 0,10 % (*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

Precipitation of sulphate as barium sulphate in dilute hydrochloric acid.

Separation of the precipitate, heating at 800 ± 25 °C and weighing.

4 REAGENTS

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

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

4.2 Sulphuric acid, ρ approximately 1,84 g/ml, about 96 % (*m/m*) or 36 N solution.

4.3 Barium chloride dihydrate ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$), 122 g/l or approximately 1 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 nitric acid solution, ρ approximately 1,40 g/ml, and dilute to 100 ml.

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

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Platinum crucible, top diameter approximately 30 mm and depth approximately 30 mm.

5.2 Electric oven, capable of being controlled at 110 ± 2 °C.

5.3 Electric furnace, capable of being controlled at 800 ± 25 °C.

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 0,01 g, a mass of the solid or liquid test sample corresponding to approximately 10 g of KOH (see ISO 2466).

6.2 Preparation of the test solution

Place the test portion (6.1) in a 600 ml beaker. 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 5 drops of the methyl orange solution (4.5) and slowly, while stirring, the volume of the hydrochloric acid solution (4.1) required for neutralization. Then add *immediately* 2 ml in excess of this acid.

Transfer the solution quantitatively to a 200 ml one-mark volumetric flask, dilute to the mark and mix.

Filter on a dry, slow-speed, ashless filter paper of diameter about 90 mm, and discard the first 10 ml of the filtrate.

6.3 Determination

Place 100,0 ml of the test solution (6.2) in a beaker of suitable capacity (for example 600 ml). Bring to the boil, stir continuously and add 10 ml of the barium chloride solution (4.3) drop by drop (the addition should take about 90 s).

Maintain boiling for 2 min, stirring all the time. Heat on a boiling water bath for 2 h; stop heating and allow to stand for about 16 h.

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Filter on an ashless slow-speed filter paper of approximately 90 mm diameter (pore diameter between 0,4 and 1 μm approximately).

Wash the precipitate onto the filter paper with hot water until 10 ml of the liquid flowing from the funnel remain clear for 5 min after the addition of 10 ml of the nitric silver nitrate solution (4.4).

Place the filter paper and its contents in the platinum crucible (5.1) previously tared after heating in the furnace (5.3) controlled at $800 \pm 25^\circ\text{C}$ and allowing to cool in a desiccator.

Place the crucible and its contents in the oven (5.2) controlled at $110 \pm 2^\circ\text{C}$ until complete desiccation. Then heat in the furnace, gently at first to char the filter paper, and then at $800 \pm 25^\circ\text{C}$ for 15 min. Allow to cool in a desiccator to ambient temperature and weigh to the nearest 0,000 1 g.

If the ignited precipitate shows a greyish colour due to the presence of graphitic carbon, add 1 drop of the sulphuric acid solution (4.2) before weighing, again place in the furnace, heat at $800 \pm 25^\circ\text{C}$ for 15 min and allow to cool in the desiccator to ambient temperature.

7 EXPRESSION OF RESULTS

The sulphate content, expressed as a percentage by mass of potassium sulphate (K_2SO_4), is given by the formula :

$$m_1 \times \frac{200}{100} \times \frac{100}{m_0} \times 0,746\ 6 = 149,32 \frac{m_1}{m_0}$$

where

m_0 is the mass, in grams, of the test portion (6.1);

m_1 is the mass, in grams, of the barium sulphate weighed;

0,747 6 is the conversion factor for BaSO_4 to K_2SO_4 .

8 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.

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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 molybdosilicate photometric method.
- ISO 996 – Determination of silica content – Gravimetric method by precipitation of quinoline molybdosilicate.
- ISO 997 – Determination of calcium – EDTA (*d*-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.

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