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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 993

POTASSIUM HYDROXIDE FOR INDUSTRIAL USE

DETERMINATION OF SULPHATE CONTENT

BARIUM SULPHATE GRAVIMETRIC METHOD

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 993, *Potassium hydroxide for industrial use – Determination of sulphate content – Barium sulphate gravimetric method,* 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. 1102) 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
Belgium
Brazil
Chile
Cuba
Czechoslovakia
Germany
Hungary
India
Ireland
Israel
Japan
Korea, Dem. P. Rep. of
Netherlands
New Zealand
Poland
Portugal
Romania
South Africa, Rep. of
Spain
Switzerland
Thailand
Turkey
U.A.R.
United Kingdom
U.S.S.R.
Yugoslavia

Three Member Bodies opposed the approval of the Draft :

France
Italy
U.S.A.

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 993 February 1969

POTASSIUM HYDROXIDE FOR INDUSTRIAL USE

DETERMINATION OF SULPHATE CONTENT

BARIUM SULPHATE GRAVIMETRIC METHOD

1. SCOPE

This ISO Recommendation describes a gravimetric method for the determination of the sulphate content in potassium hydroxide for industrial use, for contents greater than 0.10 % (m/m), expressed as potassium sulphate and calculated on KOH.

2. PRINCIPLE

Precipitation of SO_4^{2-}ions as barium sulphate in diluted hydrochloric acid. Separation of the precipitate, ignition at 800 °C and weighing.

3. REAGENTS

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

3.1 *Hydrochloric acid,* approximately N solution.

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

3.3 *Sulphuric acid,* approximately $d = 1.83$, 93.6 % (m/m) or 35 N solution.

3.4 *Barium chloride dihydrate,* 122 g/l, approximately N solution.

3.5 *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, approximately $d = 1.4$, and dilute to 100 ml.

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

4. APPARATUS

4.1 *Ordinary laboratory apparatus.*

4.2 *Platinum crucible,* approximately 30 mm in top diameter and 30 mm deep.

5. PROCEDURE

5.1 Preliminary, approximate determination of total alkalinity of the sample solution

Transfer 20.0 ml of the sample solution A* to a 500 ml conical flask. Add approximately 80 ml of water and 5 drops of the methyl orange solution (3.6) and titrate with the hydrochloric acid solution (3.1) until the yellow colour of the indicator turns to orange. Take note of the quantity of hydrochloric acid solution (3.1) used and discard the solution.

5.2 Determination

5.2.1 *Test portion.* Transfer 100.0 ml of the sample solution A* to a beaker of suitable capacity (600 ml, for example).

5.2.2 *Determination.* Acidify the solution by adding slowly a volume of hydrochloric acid solution (3.2) sufficient to neutralize it (calculate this volume by multiplying the volume, in millilitres, of the hydrochloric acid solution (3.1) used for the preliminary determination of the total alkalinity of the sample solution, as described in clause 5.1, by 0.417).

Then add an approximately 2 ml excess of hydrochloric acid solution (3.2).**

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

Filter on a dry slow-speed ashless filter paper, 90 mm in diameter, and discard the first 10 ml of filtrate. Transfer 100.0 ml of the filtrate to a beaker of suitable capacity (600 ml, for example).

Bring to the boil, stop heating and add 10 ml of the barium chloride solution (3.4) drop by drop (the addition should take about 1 minute and 30 seconds).

Bring to the boil again and allow to boil for 2 minutes. Heat on a boiling water bath for 2 hours; stop heating and allow to stand for about 16 hours.

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 minutes after the addition of 10 ml of the solution of silver nitrate (3.5). Place the filter paper and its contents in the platinum crucible (4.2), previously tared after igniting in a furnace at 800 °C and allowing to cool in a desiccator.

Dry in an oven at approximately 110 °C, then ignite in a furnace, gently at first to char the filter paper, and then at 800 °C for 15 minutes.

Allow to cool in a desiccator to room temperature and weigh.

If the ignited precipitate shows a greyish colour due to the presence of graphitic carbon, add a drop of the sulphuric acid solution (3.3) before weighing, again place in a furnace and ignite at 800 °C for 15 minutes.

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* See ISO Recommendation R 989, *Potassium hydroxide for industrial use – Preparation of sample solution*, clause 5.2.

** The final addition of acid should be carried out rather quickly so as to keep the silica in solution and prevent flocculation which may occur when the solution remains neutral or slightly alkaline for a long time. This simple precaution avoids the preliminary insolubilization of silica, which is a long and disagreeable procedure.