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Potassium hydroxide for industrial use — Determination of chlorides content — Mercurimetric method

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FOREWORD

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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.

International Standard ISO 992 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in September 1973.

It has been approved by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 992-1969, of which it constitutes a technical revision.

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Potassium hydroxide for industrial use — Determination of chlorides content — Mercurimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a mercurimetric method for the determination of the chlorides content of potassium hydroxide for industrial use.

The method is applicable to products of which the chlorides content, expressed as chlorine (Cl), is greater than 25 mg/kg.

NOTE — If 0,02 N standard volumetric mercury(II) nitrate solution is used, the method is applicable to products of which the chlorides content, expressed as chlorine (Cl), is greater than 10 mg/kg.

2 REFERENCE

ISO 2466, *Potassium hydroxide for industrial use — Sampling — Test sample — Preparation of the main solution for carrying out certain determinations.*

3 PRINCIPLE

Titration of the Cl^- ion with mercury(II) nitrate in the presence of diphenylcarbazone as indicator.

4 REAGENTS

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

4.1 Nitric acid, ρ approximately 1,40 g/ml, about 68 % (*m/m*) solution or approximately 14 N, of which the chlorides content, expressed as chlorine (Cl), is not greater than 1 mg/kg.

4.2 Nitric acid, approximately 2 N solution.

4.3 Sodium hydroxide, approximately 2 N solution.

4.4 Sodium chloride, 0,05 N standard reference solution.[1)]

Weigh, to the nearest 0,000 1 g, 2,922 1 g of sodium chloride, previously dried for 1 h at 500 °C and cooled in a desiccator. Dissolve in water in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

4.5 Standard end-point matching solution

Pour 200 ml of water into a 500 ml conical flask, add 3 drops of the bromophenol blue solution (4.7), and the nitric acid solution (4.2), drop by drop, until the colour changes from blue to yellow. Add an excess of 3 drops of this acid, 0,5 to 1,0 ml of the diphenylcarbazone solution (4.8) and, from a burette, the volume of the standard volumetric mercury(II) nitrate solution (4.6) necessary to change the colour of the solution from yellow to mauve (2 drops).

Prepare this standard solution immediately before use.

4.6 Mercury(II) nitrate, 0,05 N standard volumetric solution.

4.6.1 *Preparation of the solution*

Weigh 5,43 ± 0,01 g of mercury(II) oxide (HgO), dissolve in 10 ml of the nitric acid solution (4.1) in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

Standardize this solution following the procedure specified in 4.6.2, adjusting it to the exact concentration, if necessary.

NOTE — Analysts who can detect easily the diphenylcarbazone colour change can take advantage of a 0,02 N standard volumetric solution (2,18 g of HgO in 1 000 ml, standardized against a standard reference solution of sodium chloride containing 1,168 8 g of NaCl in 1 000 ml) in order to increase the sensitivity of the method.

4.6.2 *Standardization of the solution*

Transfer 40,0 ml of the standard reference sodium chloride solution (4.4) to a 500 ml conical flask, followed by 160 ml of water and 3 drops of the bromophenol blue solution (4.7). Add, drop by drop, the nitric acid solution (4.2) until the colour of the indicator changes from blue to yellow, then add an excess of 3 drops of this acid and a volume of the diphenylcarbazone solution (4.8) identical to that added for the standard end-point matching solution (4.5). Titrate the chloride with the mercury(II) nitrate solution to be standardized (4.6.1) until the colour matches the mauve of the standard end-point matching solution (4.5) and deduct the volume of mercury(II) nitrate solution (4.6.1) added during the preparation of this standard end-point matching solution (2 drops).

The volume corresponding to the specified concentration is 40,00 ml.

1) See note to 4.6.1.

4.7 Bromophenol blue, 1 g/l solution in 95 % (*V/V*) ethanol.

4.8 Diphenylcarbazone, 5 g/l solution in 95 % (*V/V*) ethanol.

Store this solution in a refrigerator and replace it when it no longer gives a sharp colour change.

5 APPARATUS

Ordinary laboratory apparatus.

6 PROCEDURE

6.1 Test portion

In a weighing bottle fitted with a stopper, weigh, to the nearest 0,1 g, a quantity of the solid or liquid test sample corresponding to 25 g of KOH (see ISO 2466).

6.2 Determination

6.2.1 *Preparation of the test solution*

Transfer the test portion (6.1) to a 500 ml conical flask. Add 100 ml of water and then, cautiously, 30 ml of the nitric acid solution (4.1). Cool to room temperature, add 3 drops of the bromophenol blue solution (4.7) and then the nitric acid solution (4.1) until the colour changes from blue to yellow.

Add the sodium hydroxide solution (4.3), drop by drop, until the colour changes to blue, then the nitric acid solution (4.2) until the colour turns yellow and finally an excess of 3 drops of this acid. Dilute to about 200 ml.

6.2.2 *Titration*

Add to the resultant solution (6.2.1) a volume of the diphenylcarbazone solution (4.8) identical to that added for the standard end-point matching solution (4.5) and titrate the chloride with the standard volumetric mercury(II) nitrate solution (4.6) until the colour matches the mauve of the standard end-point matching solution (4.5).

7 EXPRESSION OF RESULTS

The chlorides content, expressed as milligrams of chlorine (Cl) per kilogram, is given by the formula

$$(V_0 - V_1) \times \frac{1\,000}{m} \times 1{,}773 = \frac{1\,773\,(V_0 - V_1)}{m}$$

where

m is the mass, in grams, of the test portion;

V_0 is the volume, in millilitres, of the standard volumetric mercury(II) nitrate solution (4.6) used for the titration;

V_1 is the volume, in millilitres, of the standard volumetric mercury(II) nitrate solution (4.6) used in the preparation of the standard end-point matching solution (4.5);

1,773 is the mass, in milligrams, of chlorine (Cl) corresponding to 1 ml of the standard volumetric mercury(II) nitrate solution (4.6).

Express the result to the nearest 1 mg/kg.

NOTES

1 If 0,02 N solutions of mercury(II) nitrate and sodium chloride are used, the formula becomes

$$\frac{709{,}1\,(V_0 - V_1)}{m}$$

2 If the concentration of the standard volumetric solution used is not exactly as specified in the list of reagents, an appropriate correction should be made.

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 in the International Standard to which reference is made, or regarded as optional.