INTERNATIONAL STANDARD 3177

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

Hydroxyde de potassium à usage industriel — Dosage des chlorures — Méthode photométrique

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FOREWORD

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

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

No Member Body expressed disapproval of the document.

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

1 SCOPE AND FIELD OF APPLICATION

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

The method is applicable to products having chlorides contents, expressed as chlorine (Cl), between 2 mg/kg and 50 mg/kg.

NOTE — For contents exceeding 50 mg/kg, use the method specified in ISO 992, *Potassium hydroxide for industrial use — Determination of chlorides content — Mercurimetric method*, or reduce the volume of the test solution according to the note in 7.4.3.

2 REFERENCE

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

3 PRINCIPLE

Quantitative displacement of the thiocyanate (SCN^-) ions of the mercury(II) thiocyanate by the chloride (Cl^-) ions contained in the test portion. Reaction of the SCN^- ions with iron(III) nitrate to form iron(III) thiocyanate (red).

Photometric measurement of the colour at a wavelength of about 450 nm.

4 REACTIONS

$$2KCl + Hg(SCN)_2 \rightarrow HgCl_2 + 2KSCN$$

$$3KSCN + Fe(NO_3)_3 \rightarrow 3KNO_3 + Fe(SCN)_3$$

5 REAGENTS

The preparation and storage of the reagents, as well as the sampling and determination, shall take place in an atmosphere free from chlorine and hydrochloric acid. During the analysis, use only reagents of recognized analytical reagent grade and only double-distilled water, or water of equivalent purity.

5.1 Nitric acid, ρ approximately 1,40 g/ml, about 68 % (*m/m*) solution or approximately 14 N solution, having a chlorides content, expressed as chlorine (Cl), not exceeding 0,5 mg/kg.

5.2 Iron(III) nitrate solution corresponding to 8 g of Fe per litre.

Pour 80 ml of water into a 500 ml conical flask, and add 4,0 g of pure (99,5 % minimum) iron wire. Cautiously add 80 ml of the nitric acid solution (5.1). Heat gradually and then boil in a ventilated fume cupboard until the reaction is complete and nitrous fumes have been completely eliminated. Decolorize the solution by adding a few drops of hydrogen peroxide solution (30 % *m/m*) and boil again for a few minutes. After cooling, transfer quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

5.3 Mercury(II) thiocyanate, 0,5 g/l solution.

Weigh, to the nearest 0,001 g, 0,100 g of mercury(II) thiocyanate [$Hg(SCN)_2$] and dissolve in 180 ml of water at 50 °C, while stirring.

Filter, dilute to the mark in a 200 ml one-mark volumetric flask and mix.

Prepare this solution at the time of use.

5.4 Sodium chloride, standard solution corresponding to 0,100 g of Cl per litre.

Weigh, to the nearest 0,001 g, 0,165 g of sodium chloride, previously dried at 500 °C for 1 h and cooled in a desiccator, dissolve in water, dilute to the mark in a 1 000 ml one-mark volumetric flask and mix.

1 ml of this standard solution contains 0,1 mg of Cl.

5.5 Sodium chloride, standard solution corresponding to 10 mg of Cl per litre.

Take 20,0 ml of the sodium chloride standard solution (5.4), dilute to the mark in a 200 ml one-mark volumetric flask and mix.

1 ml of this standard solution contains 0,01 mg of Cl.

Prepare this solution at the time of use.

5.6 Phenolphthalein, 10 g/l solution in ethanol, 95 % (*V/V*).

6 APPARATUS

Ordinary laboratory apparatus, carefully rinsed with the nitric acid solution (5.1) and double-distilled water, and

6.1 Spectrophotometer, or

6.2 Photoelectric absorptiometer, fitted with filters providing maximum transmission in the neighbourhood of 450 nm.

7 PROCEDURE

7.1 Test portion

Weigh in a polyethylene beaker, to the nearest 0,01 g, a quantity of solid or liquid sample corresponding to 20 g of KOH (see ISO 2466).

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 the reagents used for the determination but omitting the test solution and the quantity of nitric acid necessary for its neutralization.

7.3 Preparation of calibration curve

7.3.1 *Preparation of the standard colorimetric solutions*, for photometric measurements with 4 or 5 cm cells.

Into a series of five 50 ml one-mark volumetric flasks, introduce the volumes of the standard sodium chloride solution (5.5) shown in the following table.

Standard sodium chloride solution (5.5)	Corresponding mass of chlorine (Cl)
ml	mg
0*	0
1,0	0,010
2,5	0,025
5,0	0,050
7,5	0,075

* Compensation solution.

Then add in turn :

- 5 ml of the nitric acid solution (5.1),
- 5 ml of the iron(III) nitrate solution (5.2), and
- 20 ml of the mercury(II) thiocyanate solution (5.3).

Dilute to the marks, mix and leave for 30 min to allow colour development.

7.3.2 *Photometric measurements*

Carry out the photometric measurements using either the spectrophotometer (6.1), at a wavelength of about 450 nm, or the photoelectric absorptiometer (6.2), fitted with suitable filters, after having adjusted the instrument to zero absorbance against the compensation solution.

7.3.3 *Preparation of the calibration chart*

Plot a graph, having, for example, the number of milligrams of chlorine (Cl) contained in 50 ml of the standard colorimetric solutions on the abscissa and the corresponding values of absorbance on the ordinate.

7.4 Determination

7.4.1 *Preparation of the test solution*

Dissolve the test portion (7.1) in, or dilute it with, water and cool to room temperature. Transfer the solution quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark and mix.

7.4.2 *Colour development*

Transfer 10,0 ml of the test solution (7.4.1) to a 50 ml one-mark volumetric flask. Add 3 drops of the phenolphthalein solution (5.6) and neutralize with the nitric acid solution (5.1) added slowly, with stirring and thorough cooling under cold running water.

Add a further 5 ml of the nitric acid solution (5.1), then 5 ml of the iron(III) nitrate solution (5.2) and 20 ml of the mercury(II) thiocyanate solution (5.3).

Dilute to the mark, mix and leave for 30 min to allow colour development.

7.4.3 *Photometric measurement*

Carry out the photometric measurement following the procedure specified in 7.3.2, after having adjusted the instrument to zero absorbance against the blank solution (7.2).

NOTE — If the absorbance exceeds the maximum of the calibration curve, repeat the determination using a smaller volume of test solution and modifying the calculation accordingly.

8 EXPRESSION OF RESULTS

By means of the calibration chart (7.3.3), determine the quantity of Cl corresponding to the value of the photometric measurement.

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

$$m_1 \times \frac{100}{10} \times \frac{1\,000}{m_0} = \frac{10\,000\, m_1}{m_0}$$

where

m_0 is the mass, in grams, of the test portion (7.1);

m_1 is the mass, in milligrams, of Cl found in the determination.

Express the result to the nearest 1 mg/kg.

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 Standards to which reference is made, or regarded as optional.

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