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Potassium hydroxide for industrial use — Determination of sodium content — Flame emission spectrophotometric 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 1550 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in September 1971.

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

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

No Member Body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R/550 – 1970.

Potassium hydroxide for industrial use – Determination of sodium content – Flame emission spectrophotometric method

1 SCOPE

This International Standard specifies a flame emission spectrophotometric method for the determination of sodium content of potassium hydroxide for industrial use.

2 FIELD OF APPLICATION

The method is applicable to products having sodium contents greater than 0,01 % (*m/m*).

3 PRINCIPLE

Dissolution of a test portion, followed by acidification to pH 1. Atomization of the resultant solution in the burner of a flame spectrophotometer.

Determination of the sodium content by measurement of the intensity of the radiation emitted at 589 nm, compared with the intensity obtained for standard sodium chloride solutions, the acidity and potassium chloride content of which are equivalent to that of the test solution.

4 REAGENTS

4.1 Water. Distilled water, or water of equivalent purity, shall be used in the test. The sodium content of this water shall not exceed 0,02 mg/l. Confirm this value by the following test :

The spectrophotometer having been adjusted so that a zero reading is obtained (minimum of the scale) for water (4.1) and about 100 (maximum of the scale) for a standard solution containing 10 mg/l of sodium, the reading obtained for the water, evaporated in a sodium-free vessel (platinum, silica, etc.) until it is reduced to one-fiftieth of its initial volume, shall not exceed 10.

4.2 Hydrochloric acid, approximately 2 N solution, obtained by dilution of a hydrochloric acid solution ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution or approximately 12 N, of which the sulphated ash is less than 10 mg/kg (corresponding to a maximum sodium content of 3,2 mg/kg of Na.

4.3 Potassium chloride, having a sodium content of less than 0,02 % (*m/m*), dried for 4 h at 500 °C then cooled in a desiccator.

Such a product may be obtained commercially or may be prepared in the laboratory by the following method :

Dissolve 10 g of analytical reagent grade potassium chloride in 20 ml of water, warming it slightly. Immediately add 230 ml of pure 1-methylpropan-1-ol. Stir for a few moments with a glass rod and leave to stand for 2 min. Filter through a sintered glass crucible, carrying the precipitate down with 1-methylpropan-1-ol. Wash, on the filter, with 1-methylpropan-1-ol. Dry the precipitate at 500 °C for 4 h.

4.4 Standard sodium chloride solution, corresponding to 1,000 g of sodium per litre.

Weigh, to the nearest 0,001 g, 2,543 g of sodium chloride previously dried for 4 h at 500 °C and then cooled in a desiccator. Dissolve it in the water (4.1), dilute to 1 000 ml in a one-mark volumetric flask and mix.

1 ml of this standard solution contains 1 mg of Na.

4.5 Potassium chloride, 20 g/l solution, prepared from potassium chloride (4.3) and water (4.1).

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

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Flame spectrophotometer, fitted with an atomizer-burner, fed with a butane/air mixture in such proportions that combustion is complete.

After a warming up period of 30 to 60 min to ensure that the apparatus is in normal operation, the readings shall be reproducible and stable 20 to 40 s after the start of atomization.

6 PROCEDURE

6.1 Preparation of the calibration curve

6.1.1 Preparation of the standard matching solutions

Transfer to a series of twelve 1 000 ml one-mark volumetric flasks, 100 ml of the potassium chloride solution (4.5) and 50 ml of the hydrochloric acid solution (4.2), then add the quantities of the sodium chloride standard solution (4.4), indicated in the following table :

Sodium chloride standard solution (4.4)	Corresponding mass of Na
ml	mg
0*	0
3,0	3
6,0	6
9,0	9
12,0	12
15,0	15
18,0	18
21,0	21
24,0	24
27,0	27
30,0	30
32,0	32

* Blank test on the calibration curve reagents.

Dilute to the mark with the water (4.1) and mix.

The acidity of these standard matching solutions is approximately 0,1 N.

NOTE — These concentrations may be adjusted, if necessary, according to the characteristics of the apparatus used, so that the measurements may be taken in the highest sensitivity range of the equipment used.

6.1.2 Spectrophotometric measurements

Switch on the spectrophotometer (5.1) in advance to allow sufficient time for its stabilization. Adjust the sensitivity of the apparatus and the opening of the slit according to the characteristics of the apparatus and to ensure a band pass of not more than 6 nm, centred on the emission maximum (theoretical value 589 nm).

Atomize the standard matching solutions (6.1.1) in turn in the centre of the flame and measure the intensity of the radiation emitted in each case.

Care should be taken to keep the rate of atomization constant throughout the measurements.

6.1.3 Preparation of the calibration chart

Plot a graph having, for example, the sodium contents as abscissae and as ordinates, on a logarithmic scale, the corresponding values of the luminous intensities emitted by the standard matching solutions, deducted from the value measured for the zero term (blank test on the calibration curve reagents).

NOTE — The use of this curve leads to only approximate results. It is particularly advisable to carry out a preliminary test and to deduce from this the approximate sodium content of the test solution. In order to obtain more precise results, it is advisable to proceed by interpolation, bracketing the determination of the solution under examination between two determinations carried out on standard matching solutions of sufficiently close sodium content.

6.2 Determination

6.2.1 Test portion

With the aid of a pipette, take 40,0 ml of the main solution A* containing a mass of test sample corresponding to approximately 50 g of KOH per 1 000 ml.

6.2.2 Preparation of the test solution

Transfer the test portion (6.2.1) to a 1 000 ml one-mark volumetric flask, add about 200 ml of water (4.1), 5 drops of the methyl orange solution (4.6) and the hydrochloric acid solution (4.2) until the indicator changes colour. Add an excess of 50 ml of this acid, dilute to the mark and mix.

6.2.3 Spectrophotometric measurements

6.2.3.1 PRELIMINARY MEASUREMENT

Carry out a preliminary measurement on the test solution (6.2.2), according to the procedure specified in 6.1.2. Deduce the approximate sodium content of the test solution, by reference to the calibration curve.

6.2.3.2 BRACKETING MEASUREMENT

Carry out a second measurement on the test solution (6.2.2) by bracketing it between two standard matching solutions (6.1.1) differing in sodium content by only 3 mg/l.

NOTE — If the sample contains more sodium than had been anticipated, it is advisable to extend the range of standard matching solutions to include some of higher sodium content.

* See ISO 2466, *Potassium hydroxide for industrial use — Sampling — Test sample — Preparation of the main solution for carrying out certain determinations.* (At present at the stage of draft.)

7 EXPRESSION OF RESULTS

7.1 The concentration, C , of sodium expressed in milligrams per litre, of the test solution (6.2.2), is given by the formula

$$C = C_1 + (C_2 - C_1) \frac{E - E_1}{E_2 - E_1}$$

where

C_1 is the concentration, in milligrams per litre, of the weaker matching solution used for the determination;

C_2 is the concentration, in milligrams per litre, of the stronger standard matching solution used for the determination;

E is the value of the measurement corresponding to the test solution.;

E_1 is the value of the measurement corresponding to C_1 ;

E_2 is the value of the measurement corresponding to C_2 .

7.2 The sodium (Na) content of the test sample is given, as a percentage by mass, by the formula

$$C \times \frac{1}{1\ 000} \times \frac{1\ 000}{40} \times \frac{100}{m} = \frac{2,5\ C}{m}$$

where

m is the mass, in grams, of the test portion used for the preparation of the main solution A.

C is the concentration of sodium (Na) in the test solution (6.2.2) expressed in milligrams per litre (see 7.1).

Express the result to the second decimal place.

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.

