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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1550

POTASSIUM HYDROXIDE FOR INDUSTRIAL USE

DETERMINATION OF SODIUM

FLAME EMISSION PHOTOMETRY METHOD

1st EDITION

July 1970

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 1550, *Potassium hydroxide for industrial use – Determination of sodium – Flame emission photometry 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 to the adoption of Draft ISO Recommendation No. 1550 which was circulated to all the ISO Member Bodies for enquiry in July 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

- Austria
- Belgium
- Colombia
- Czechoslovakia
- Germany
- Hungary
- India
- Iran
- Israel
- Italy
- Korea, 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.

The following Member Body opposed the approval of the Draft :

France

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 1550 July 1970

POTASSIUM HYDROXIDE FOR INDUSTRIAL USE

DETERMINATION OF SODIUM

FLAME EMISSION PHOTOMETRY METHOD

1. SCOPE

This ISO Recommendation describes a method of determining, by flame emission photometry, the sodium in potassium hydroxide for industrial use (solid or dissolved product).

2. FIELD OF APPLICATION

The method can be applied to the determination of sodium contents (expressed as NaOH) which are greater than or equal to 0.1 % (m/m) (based on KOH).

3. PRINCIPLE

Neutralization of the test portion with hydrochloric acid and atomization of the solution in the burner of a flame spectrophotometer.

Determination of the Na^+ ions content by measuring the intensity of the emission at 589 nm and comparing it with the values obtained for standard matching solutions of sodium chloride containing known quantities of potassium chloride.

4. REAGENTS

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

4.1 *Hydrochloric acid*, 2 N solution approximately, free from sodium.

4.2 *Potassium chloride*, free from sodium, dried for 4 hours at 500 °C and then cooled in a desiccator.

NOTE. – Such a product is commercially available; however it can be prepared in the laboratory as follows : heating gently, dissolve 10 g of potassium chloride, reagent grade, in 20 ml of water and add immediately 230 ml of pure isopropanol. Stir with a glass rod, then allow to rest for 2 minutes. Filter on a sintered glass crucible, removing the precipitate by means of isopropanol. Wash the precipitate in the filter with isopropanol and dry it at 500 °C for 4 hours.

4.3 *Sodium chloride*, standard solution corresponding to 0.400 g/l of NaOH. In a beaker of suitable capacity (for example 100 ml) weigh to within 0.2 mg, 0.2922 g of sodium chloride (corresponding to 0.200 g of NaOH) which has been previously dried for 4 hours at 500 °C and then cooled in a desiccator. Dissolve in a little water, transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix. 1 ml of this solution corresponds to 0.4 mg of NaOH.

4.4 *Phenolphthalein*, 10 g/l ethanolic solution.
Dissolve 1 g of phenolphthalein in 95 % (V/V) ethanol and dilute to 100 ml with the same ethanol.

5. APPARATUS

Ordinary laboratory apparatus and

5.1 *Flame spectrophotometer* provided with an atomizer-burner, adjusted in such a way as to cause the emission of the spectral line of sodium at 589 nm.

6. PROCEDURE

6.1 Preparation of standard matching solutions

In a series of four 250 ml one-mark volumetric flasks place :

- initially 1.302, 1.316, 1.322 and 1.328 g of potassium chloride (4.2),
- then 50.0, 25.0, 12.50 and 2.50 ml of the sodium chloride standard solution (4.3).

Add 50 ml of water,then, after dissolution of the potassium chloride, dilute to the mark and mix thoroughly.

Transfer 100.0 ml of each solution to 250 ml one-mark volumetric flasks and dilute to the mark.

The standard matching solutions, thus obtained, contain :

8, 4, 2 and 0.4 mg of NaOH for each 250 ml solution respectively (i.e. 2, 1, 0.5 and 0.1 % (m/m) referred to potassium hydroxide).

6.2 Determination

6.2.1 *Test portion.* Into a beaker of suitable capacity (for example 400 ml) weigh, to the nearest 0.01 g, a mass of the test sample (solid or dissolved product) containing 10 ± 0.1 g of KOH.

6.2.2 *Preparation of the sample solution.* In the case of a solid product dissolve the test portion (6.2.1) in about 50 ml of water and cool to room temperature. Transfer the solution quantitatively into a 250 ml one-mark volumetric flask.

In the case of a dissolved product, transfer the test portion (6.2.1) directly and quantitatively to a 250 ml one-mark volumetric flask. Add 1 drop of the phenolphthalein solution (4.4) and then neutralize the potassium hydroxide by the addition of the hydrochloric acid solution (4.1), until the solution turns colourless.

Cool to room remperature, dilute to the mark and mix.

Transfer 10.00 ml of this solution to a 250 ml one-mark volumetric flask, dilute to the mark and mix thoroughly. This solution constitutes the sample solution.

6.2.3 *Photometric measurements.* Turn on the apparatus (5.1) in advance in order to allow for the period required for its stabilization.

Adjust the sensitivity of the apparatus and the aperture of the slit to ensure a maximum width of the pass band of 6 nm, centred on the emission maximum (theoretical value 589 nm). Atomize the various standard matching solutions (6.1) and the sample solution (6.2.2) in the heart of the flame, and measure the intensity of the lines emitted.

Ensure that the rate at which the solutions are atomized in the flame is constant throughout the measurement period.

6.2.4 *Preparation of the calibration chart and calculation of the results.* Plot a graph indicating on the X-axis the NaOH content of the standard matching solution expressed in milligrammes/totality and on the Y-axis, the corresponding values of the luminous intensities emitted by the standard matching solutions, using a logarithmic scale.

Plot on the curve obtained the luminous intensity emitted by the sample solution and deduce therefrom the NaOH content of the sample solution expressed in milligrammes/totality (A).

In order to obtain a more precise value for A it is useful to bracket the sample solution between two standard matching solutions of very similar concentrations, which are obtained by suitable mixing of the standard matching solutions (6.1).