INTERNATIONAL STANDARD 981

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Sodium hydroxide for industrial use — Determination of chloride 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 981 (originally Draft No. 2458) was drawn up by Technical Committee ISO/TC 47, *Chemistry*.

It was approved in January 1972 by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 981-1969, *Sodium hydroxide for industrial use — Determination of chloride content — Volhard volumetric method.*

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Sodium hydroxide for industrial use — Determination of chloride content — Mercurimetric method

1 SCOPE

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

2 FIELD OF APPLICATION

The method is applicable to products of which the sodium chloride content is greater than 0,005 % (*m/m*).

NOTE — If 0,02 N standard volumetric mercury(II) nitrate is used, the method is applicable to products of which the sodium chloride content is greater then 0,002 % (*m/m*).

3 PRINCIPLE

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

4 REAGENTS

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

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

4.2 Nitric acid, approximately 2 N solution.

4.3 Sodium hydroxide, 7,5 % (*m/m*) or approximately 2 N solution.

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

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

4.5 Standard end-point matching solution

Prepare this standard solution immediately before use. Pour 200 ml of water into a 500 ml conical flask, add 3 drops of the bromophenol blue solution (4.7), and then add 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).

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) and dissolve it 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 described 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 sodium chloride standard reference 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 the mercury(II) nitrate solution (4.6.1) added during the preparation of this standard end-point matching solution (2 drops).

The correct amount is 40,00 ml.

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

1) See note to 4.6.1.

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 20 g of NaOH.

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), then add 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 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).

NOTE – Different operating conditions for standardizing the mercury(II) nitrate solution and for making the determination are only justified in cases where the chloride content to be determined is low. In other cases this would not be so, and the standardization and the determination should then be carried out in conditions as nearly alike as possible.

7 EXPRESSION OF RESULTS

The chloride content, expressed as sodium chloride (NaCl), is given, as a percentage by mass, by the formula

$$(V_0 - V_1) \times \frac{100}{m} \times 0{,}002\,922 = \frac{0{,}292\,2\,(V_0 - V_1)}{m}$$

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);

0,002 922 is the mass, in grams, of sodium chloride corresponding to 1 ml of the standard volumetric mercury(II) nitrate solution (4.6).

Express the result to two places of decimals.

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

$$\frac{0{,}116\,9\,(V_0 - V_1)}{m}$$

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 regarded as optional.