BRITISH STANDARD

BS 6075-2:
1981
ISO 981:1973

Methods of

Sampling and test for sodium hydroxide for industrial use —

Part 2: Determination of chloride content (mercurimetric method)

[ISO title: Sodium hydroxide for industrial use — Determination of chloride content — Mercurimetric method]

NOTE It is recommended that this Part be read in conjunction with the information in the "*General introduction*" published separately as BS 6075-0.

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The following BSI reference relates to the work on this standard:
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Amendments issued since publication

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Contents

National foreword

This Part of BS 6075 is identical with ISO 981 "*Sodium hydroxide for industrial use — Determination of Chloride content — Mercurimetric method*", published in 1973 by the International Organization for Standardization (ISO).

The National appendix A to this Part of this standard describes a procedure to remove mercury from waste solutions from the analytical procedures described herein. This procedure is technically equivalent to that described in ISO 5790 "*Inorganic chemical products for industrial use — General method for determination of chloride content — Mercurimetric method*" (which has not been implemented as a British Standard).

Terminology and conventions. The text of the International Standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is especially drawn to the following.

The comma has been used throughout as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Wherever the words "International Standard" appear, referring to this standard, they should be read as "British Standard".

Additional information

Water. Water complying with the requirements of clause **4** is specified in BS 3978 *"Water for laboratory use"*.

Reagents. The reagents used shall be of recognized analytical quality.

Test portion. For some grades of sodium hydroxide it may be necessary to adjust the mass of the test portion (see **6.1**) to ensure that the amount of chloride present in the test solution complies with the requirements for the test method. In BS 4130 *"Sodium hydroxide (technical grades)"* details of a suitable mass for the grades specified are given.

WARNING. Mercury (II) nitrate, specified in **4.6**, is toxic (with danger of cumulative effects) and is irritant, particularly in the form of dust. Avoid breathing dust. Avoid contact with skin and eyes. Avoid pollution of waste water with mercury from waste solutions using, for example, the procedure for removal of mercury described in the National appendix A to this Part of this standard.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 4, an inside back cover and back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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.

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.

[1)] See note to **4.6.1**.

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.

National appendix A Removal of mercury from waste solutions

A.1 Scope

This National appendix A describes a method for removing mercury from waste solutions in order to prevent pollution of waste water.

It is recommended principally for waste solutions from titrations using mercury (II) nitrate solution but may be used for other solutions containing mercury.

A.2 Principle

The mercury in the collected waste solution is precipitated by reaction with alkaline sodium sulphide. Hydrogen peroxide is added to oxidize excess sodium sulphide (to prevent formation of soluble mercury polysulphides), the insoluble mercury salts are removed by decantation and filtration and the mercury-free waste solution is discarded.

A.3 Reagents

The following reagents to be used shall be of technical quality.

A.3.1 *Disodium sulphide nonahydrate,* ($Na_2S.9H_2O$).

A.3.2 *Hydrogen peroxide,* approximately 30 % (*m*/*m*) solution.

A.3.3 *Sodium hydroxide,* approximately 400 g/l solution.

A.4 Apparatus

The following apparatus is required.

A.4.1 *Polyethylene container*. A container of 50 litres capacity is suitable.

A.4.2 *Small laboratory pump or water pump,* with a suction tube the end of which is fitted with a sintered glass disc, grade No 4 (maximum pore diameter 5 μm to 15 μm) complying with the requirements of BS 1752 "*Laboratory sintered or fritted filters"* or a funnel containing cotton wool.

A.5 Procedure

Pour the waste solutions into the container (**A.4.1**).

When about 40 litres of solution has been collected, add successively, mixing by means of compressed air, 400 ml of the sodium hydroxide solution (**A.3.3**), 100 g of the sodium sulphide (**A.3.1**) and, after 10 min, slowly, 400 ml of the hydrogen peroxide solution (**A.3.2**).

Allow to stand for 24 h, then draw off the clear liquid using the pump (**A.4.2**) and discard as drain water.

Rinse the container several times with sufficient water to transfer the insoluble residue to another container reserved for its storage and subsequent disposal in a manner suitable for material containing mercury.

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Publications referred to

See national foreword.

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