BRITISH STANDARD

BS 6075-12: 1981

Methods of

Sampling and test for sodium hydroxide for industrial use —

Part 12: Determination of copper content

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

Confirmed January 2011

UDC 661.322.1:546.33 – 36:543.42.062:546.56

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 and 2, an inside back cover and a 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.

This British Standard, having been prepared under the direction of the Chemicals Standards Committee, was published under the authority of the Executive Board and comes into effect on 27 February 1981

The following BSI reference relates to the work on this standard:
Committee reference CIC/22

ISBN 0 580 12062 7

Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

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1 Scope

This Part of BS 6075 describes a method of test for the determination of the copper content of sodium hydroxide for industrial use.

The method is applicable to products having copper contents, expressed as Cu, in the ranges 0.5 mg/kg to 10 mg/kg and 0.25 mg/kg to 5 mg/kg for the solid and liquid products, respectively.

There is no corresponding International Standard for this method.

2 References

The titles of the publications referred to in this standard are listed on the inside back cover.

3 Principle

The copper present is reduced with ascorbic acid and a violet coloured complex is formed by addition of 2,2′-biquinolyl. This complex is extracted with amyl alcohol and its colour measured spectrophotometrically.

4 Reagents

The reagents used shall be of recognized analytical quality. Water complying with the requirements of BS 3978 shall be used throughout.

4.1 *Sodium hydroxide*

4.2 *Sodium sulphate,* anhydrous.

4.3 *Hydrochloric acid solution, ρ* approximately 1.18 g/ml, approximately 36 % (*m*/*m*) solution or approximately 11N.

4.4 *Amyl alcohol*

4.5 *(+)–Tartaric acid,* 500 g/l solution.

4.6 *Sodium hydroxide,* 200 g/l solution.

4.7 *L-Ascorbic acid,* 100 g/l solution, freshly prepared.

4.8 2,2′-*Biquinolyl,* 0.5 g/l solution. Dissolve 0.25 g of 2,2′-biquinolyl in the amyl alcohol (**4.4**) and dilute with more of the amyl alcohol to 500 ml.

4.9 *Bromine water,* saturated solution.

4.10 *Copper,* standard solution corresponding to 0.1 g of copper per litre. Dissolve 0.3928 g of copper (II) sulphate pentahydrate ($CuSO_4.5H_2O$) in water in a 1 000 ml one-mark volumetric flask, add 25 ml of approximately 6N sulphuric acid solution, dilute to the mark and mix.

1 ml of this standard solution contains 0.100 mg of Cu.

4.11 *Copper,* standard solution corresponding to 0.01 g of copper per litre. Dilute 10.0 ml of the standard copper solution (**4.10**) to the mark in a 100 ml one-mark volumetric flask and mix.

1 ml of this standard solution contains 10 μg of Cu.

4.12 *Narrow range indicator papers,* covering the range pH 5.5 to pH 7.0.

4.13 *Methyl orange indicator,* 0.5 g/l aqueous solution.

5 Apparatus

Ordinary laboratory apparatus and the following are required.

5.1 *Spectrophotometer,* or

5.2 *Photoelectric absorptiometer,* fitted with filters providing maximum transmission at a wavelength of about 545 nm.

5.3 *Optical cells,* 4 cm optical path length.

6 Procedure

6.1 Test portion. Weigh, to the nearest 0.01 g in a plastics weighing bottle fitted with a cover, an amount of the sample corresponding to about 10 g of sodium hydroxide.

6.2 Blank test. Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of reagents as specified in **6.4** but using 10 g of the sodium hydroxide (**4.1**).

6.3 Preparation of the calibration graph

6.3.1 *Preparation of standard matching solutions.* Into each of a series of six 500 ml stoppered separating funnels, introduce the volumes of the standard copper solution (**4.11**) shown in Table 1.

Table 1 — Volumes of standard copper solution

Standard copper solution (4.11)	Corresponding mass of copper (Cu)
ml	μg
0[a]	0
2.0	20
4.0	40
6.0	60
8.0	80
10.0	100

[a] Compensation solution.

6.3.2 *Colour development.* Treat the contents of each funnel (**6.3.1**) as follows.

Dilute with water to approximately 400 ml, then add 2 ml of the tartaric acid solution (**4.5**). Adjust the pH of the solution to about 6.0 by addition of the sodium hydroxide solution (**4.6**) using the narrow range indicator paper (**4.12**) externally. Add 2 ml of the ascorbic acid solution (**4.7**), shake so as to mix thoroughly and allow to stand for 5 min. Add 10 ml of the 2,2′-biquinolyl solution (**4.8**) and shake well for about 2 min. Extract the copper complex with two 20 ml portions of the amyl alcohol (**4.4**), and transfer the extracts to a 100 ml beaker. Add about 2 g of the anhydrous sodium sulphate (**4.2**) to the combined extracts and stir thoroughly to remove traces of water. Filter the dried extract into a 50 ml one-mark volumetric flask, wash the residual sodium sulphate twice with 2 ml portions of the amyl alcohol (**4.4**). Transfer the washings to the flask, dilute to the mark with the amyl alcohol (**4.4**) and mix.

6.3.3 *Photometric measurements.* Carry out the photometric measurements either with the spectrophotometer (**5.1**) at the wavelength of maximum absorption (about 545 nm), or with the photoelectric absorptiometer (**5.2**), fitted with suitable filters, after having adjusted the instrument to zero absorbance against the amyl alcohol (**4.4**).

6.3.4 *Plotting the calibration graph.* Deduct the absorbance of the compensation solution (**6.3.1**) from those of the standard matching solutions (**6.3.1**). Plot a graph having, for example, the copper content expressed in micrograms per 50 ml of standard matching solution, as abscissae and the corresponding values of absorbance as ordinates.

6.4 Determination

6.4.1 *Preparation of the test solution.* Transfer the test portion quantitatively to a 400 ml beaker. Add about 100 ml of water and 1 drop of the methyl orange indicator solution (**4.13**). Neutralize the solution with the hydrochloric acid solution (**4.3**), then add 5 ml in excess and add 10 ml of the bromine water (**4.9**). Boil the solution until free from bromine and allow to cool. Transfer the contents of the beaker quantitatively to a 500 ml separating funnel fitted with a stopper.

6.4.2 *Colour development.* Treat the test solution in the separating funnel (**6.4.1**) as specified in **6.3.2**.

6.4.3 *Photometric measurement.* Carry out the photometric measurement on the test solution (**6.4.2**) and on the blank test solution (**6.2**) following the procedure described in **6.3.3**, after having adjusted the instrument to zero absorbance against the amyl alcohol (**4.4**).

NOTE If the absorbance exceeds the maximum of the calibration graph, repeat the determination using a smaller amount of the test portion (**6.1**) and modifying the calculation accordingly.

7 Expression of results

By means of the calibration graph (**6.3.4**), determine the quantity of copper (Cu) corresponding to the value of the photometric measurement.

The copper content, expressed as milligrams of copper (Cu) per kilogram, is given by the following formula:

$$\frac{m_1 - m_2}{1000} \times \frac{1000}{m_0} = \frac{(m_1 - m_2)}{m_0}$$

where

m_0 is the mass of the test portion (in g)

m_1 is the mass of copper in the test solution (in μg)

m_2 is the mass of copper in the blank test solution (in μg)

Publications referred to

BS 1647, *pH scale.*
BS 3978, *Water for laboratory use.*

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