Licensed copy: Lee Shau Kee Library, HKUST, Version correct as of 03/01/2015, (c) The British Standards Institution 2013

BRITISH STANDARD

BS 6075-3:
1981
ISO 983:1974

Methods of

Sampling and test for sodium hydroxide for industrial use —

Part 3: Determination of iron content

[ISO title: Sodium hydroxide for industrial use — Determination of iron content — 1,10-Phenanthroline photometric 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.

UDC 661.322.1:546.33 – 36:543.42.062:546.72

NO COPYING WITHOUT BSI PERMISSION EXCEPT AS PERMITTED BY COPYRIGHT LAW

Licensed copy: Lee Shau Kee Library, HKUST, Version correct as of 03/01/2015, (c) The British Standards Institution 2013

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

© BSI 10-1999

The following BSI references relate to the work on this standard:
Committee reference CIC/22
Draft for comment 73/54863 DC

ISBN 0 580 12053 8

Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

Licensed copy: Lee Shau Kee Library, HKUST, Version correct as of 03/01/2015, (c) The British Standards Institution 2013

Licensed copy: Lee Shau Kee Library, HKUST, Version correct as of 03/01/2015, (c) The British Standards Institution 2013

National foreword

This Part of BS 6075 is identical with ISO 983 "*Sodium hydroxide for industrial use — Determination of iron content — 1,10-Phenanthroline photometric method*", published in 1974 by the International Organization for Standardization (ISO).

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".

Cross-reference

International Standard	Corresponding British Standard
	BS 6075 *Methods of sampling and test for sodium hydroxide for industrial use*
ISO 3195:1975	Part 5:1981 *Sampling and preparation of main test solution* (Identical)

Additional information

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

Hydrochloric acid, ρ approximately 1.18 g/ml, which is the corresponding reagent normally obtainable in the UK, is suitable for use in place of the solution specified in **4.1**.

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 iron 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 solid grades specified are given.

WARNING. Hydroxylammonium chloride, specified in **4.3**, is toxic, corrosive and irritant. Avoid contact with eyes and skin.

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 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.

© BSI 10-1999

Licensed copy: Lee Shau Kee Library, HKUST, Version correct as of 03/01/2015, (c) The British Standards Institution 2013

1 Scope and field of application

This International Standard specifies a 1,10-phenanthroline photometric method for the determination of the iron content of sodium hydroxide for industrial use.

This method is more sensitive and more widely used than the 2,2′-bipyridyl method which was specified in ISO/R 983:1969.

The method is applicable to products having iron contents equal to or greater than 0,5 mg/kg.

2 Reference

ISO 3195, *Sodium hydroxide for industrial use — Sampling — Test sample — Preparation of the main solution for carrying out certain determinations*[1].

3 Principle

Reduction of the trivalent iron by hydroxylammonium chloride, followed by the formation of a bivalent iron/1,10-phenanthroline complex in a buffered system. Photometric measurement of the coloured complex at a wavelength of about 510 nm.

4 Reagents

During the analysis, use only reagents of recognized analytical reagent grade, and only distilled water, or water of equivalent purity.

4.1 *Hydrochloric acid,* ρ approximately 1,19 g/ml, about 38 % (*m*/*m*) solution or approximately 12 N.

4.2 *Ammonia solution,* ρ approximately 0,91 g/ml, about 25 % (*m*/*m*) NH_3 solution or approximately 13 N, with a maximum iron content of 0,2 mg/kg.

4.3 *Hydroxylammonium chloride* ($NH_2OH.HCl$), 10 g/l solution.

4.4 *Buffer solution,* pH 4,9.

Dissolve 272 g of sodium acetate trihydrate ($CH_3COONa.3H_2O$) in about 500 ml of water. Add 240 ml of glacial acetic acid (ρ approximately 1,05 g/ml, 99 to 100 % (*m*/*m*) solution or approximately 17,4 N) to the solution, dilute to 1 000 ml and mix.

4.5 *Bromine water,* saturated at room temperature.

4.6 *1,10-phenanthroline hydrochloride monohydrate* ($C_{12}H_8N_2.HCl.H_2O$), 2,5 g/l solution.

This reagent may be replaced by 1,10-phenanthroline monohydrate ($C_{12}H_8N_2.H_2O$), 2,5 g/l solution.

4.7 *Iron,* standard solution, corresponding to 0,200 g of Fe per litre.

Dissolve 1,404 3 g of ammonium iron(II) sulphate hexahydrate [$(NH_4)_2Fe(SO_4)_2.6H_2O$], weighed to the nearest 0,000 1 g, in 200 ml of water. Add 20 ml of sulphuric acid, ρ approximately 1,84 g/ml, cool to room temperature, dilute to the mark in a 1 000 ml one-mark volumetric flask and mix.

4.8 *Iron,* standard solution corresponding to 0,010 g of Fe per litre.

Transfer 25,0 ml of the standard iron solution (**4.7**) to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution immediately before use.

1 ml of this standard solution contains 0,010 mg of Fe.

4.9 *Methyl orange,* 0,5 g/l solution.

5 Apparatus

Ordinary laboratory apparatus and

5.1 *Spectrophotometer,* or

5.2 *Photoelectric absorptiometer,* fitted with filters giving a maximum transmission between 500 and 520 nm.

6 Procedure

6.1 Test portion

Weigh, to the nearest 0,01 g, in a weighing bottle fitted with a ground glass lid, a mass of the test sample,[2] solid or liquid, containing between 25 and 30 g of NaOH.

6.2 Blank test

Pour 25 ml of water and a volume of the hydrochloric acid solution (**4.1**) identical to that used to neutralize the test portion (see **6.4.1**) into a 600 ml beaker. Add 40 ml of the ammonia solution (**4.2**) and 5 drops of the methyl orange solution (**4.9**) and then neutralize with the ammonia solution (**4.2**). Add the hydrochloric acid solution (**4.1**), drop by drop, until the colour changes to red, and then an excess of 2 ml of this acid. Add 5 ml of the bromine water (**4.5**) to remove the colour of the indicator, boil for 5 min, cool to room temperature, transfer the solution quantitatively to a 250 ml one-mark volumetric flask, dilute to the mark and mix. Proceed as described in **6.4.2**.

[1] At present at the stage of draft.

[2] See ISO 3195.

Licensed copy: Lee Shau Kee Library, HKUST, Version correct as of 03/01/2015, (c) The British Standards Institution 2013

6.3 Preparation of the calibration curve

6.3.1 *Preparation of the standard colorimetric solutions,* for photometric measurements with a 5 cm cell.

Into a series of five 100 ml one-mark volumetric flasks, transfer the quantities of the standard iron solution (**4.8**) indicated in the following table:

Standard iron solution (4.8)	Corresponding mass of Fe
ml	mg
0[a]	0
2,5	0,025
5,0	0,050
10,0	0,100
15,0	0,150
[a] Compensation solution	

Add 0,5 ml of the hydrochloric acid solution (**4.1**) and the amount of water necessary to make up the volume to about 50 ml to each flask. Then add 5 ml of the hydroxylammonium chloride solution (**4.3**), 5 ml of the 1,10-phenanthroline hydrochloride solution (**4.6**) and 25 ml of the buffer solution (**4.4**).

Dilute to the mark, mix and wait for 10 min.

6.3.2 ***Photometric measurements***

Carry out the photometric measurements with the spectrophotometer (**5.1**), at a wavelength of about 510 nm, or with the photoelectric absorptiometer (**5.2**), fitted with suitable filters, after having adjusted, in each case, the instrument to zero absorbance against the compensation solution.

6.3.3 ***Plotting the calibration curve***

Prepare a chart having, for example, the iron (Fe) contents in milligrams per 100 ml of the standard colorimetric solutions 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 (**6.1**) to a 1 000 ml beaker. Add 120 ml of water, and neutralize cautiously with the hydrochloric acid solution (**4.1**) in the presence of 5 drops of the methyl orange solution (**4.9**). Add an excess of 2 ml of this acid, followed by 5 ml of the bromine water (**4.5**). Boil for 5 min, cool to room temperature, transfer quantitatively to a 250 ml one-mark volumetric flask, dilute to the mark and mix.

6.4.2 ***Colour development***

Transfer 50,0 ml of the test solution (**6.4.1**) to a 100 ml one-mark volumetric flask. Add 5 ml of the hydroxylammonium chloride solution (**4.3**), 5 ml of the 1,10-phenanthroline hydrochloride solution (**4.6**) and 25 ml of the buffer solution (**4.4**). Dilute to the mark, mix and wait for 10 min.

6.4.3 ***Photometric measurement***

Measure the absorbance of the solution (**6.4.2**), as described in **6.3.2**, after having adjusted the instrument to zero absorbance against the blank test solution (**6.2**).

7 Expression of results

By reference to the calibration curve (**6.3**), determine the quantity of Fe corresponding to the value of the absorbance measured.

The iron content, expressed as milligrams of iron (Fe) per kilogram, is given by the formula

$$m_1 \times \frac{250}{50} \times \frac{1\,000}{m_0} = \frac{5\,000\,m_1}{m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in milligrams, of Fe found in the aliquot portion of the test solution.

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.

© BSI 10-1999

Publications referred to

See national foreword.

Licensed copy: Lee Shau Kee Library, HKUST, Version correct as of 03/01/2015, (c) The British Standards Institution 2013

© BSI 10-1999

Licensed copy: Lee Shau Kee Library, HKUST, Version correct as of 03/01/2015, (c) The British Standards Institution 2013

BSI — British Standards Institution

BSI is the independent national body responsible for preparing British Standards. It presents the UK view on standards in Europe and at the international level. It is incorporated by Royal Charter.

Revisions

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions.

It is the constant aim of BSI to improve the quality of our products and services. We would be grateful if anyone finding an inaccuracy or ambiguity while using this British Standard would inform the Secretary of the technical committee responsible, the identity of which can be found on the inside front cover. Tel: 020 8996 9000. Fax: 020 8996 7400.

BSI offers members an individual updating service called PLUS which ensures that subscribers automatically receive the latest editions of standards.

Buying standards

Orders for all BSI, international and foreign standards publications should be addressed to Customer Services. Tel: 020 8996 9001. Fax: 020 8996 7001.

In response to orders for international standards, it is BSI policy to supply the BSI implementation of those that have been published as British Standards, unless otherwise requested.

Information on standards

BSI provides a wide range of information on national, European and international standards through its Library and its Technical Help to Exporters Service. Various BSI electronic information services are also available which give details on all its products and services. Contact the Information Centre. Tel: 020 8996 7111. Fax: 020 8996 7048.

Subscribing members of BSI are kept up to date with standards developments and receive substantial discounts on the purchase price of standards. For details of these and other benefits contact Membership Administration. Tel: 020 8996 7002. Fax: 020 8996 7001.

Copyright

Copyright subsists in all BSI publications. BSI also holds the copyright, in the UK, of the publications of the international standardization bodies. Except as permitted under the Copyright, Designs and Patents Act 1988 no extract may be reproduced, stored in a retrieval system or transmitted in any form or by any means – electronic, photocopying, recording or otherwise – without prior written permission from BSI.

This does not preclude the free use, in the course of implementing the standard, of necessary details such as symbols, and size, type or grade designations. If these details are to be used for any other purpose than implementation then the prior written permission of BSI must be obtained.

If permission is granted, the terms may include royalty payments or a licensing agreement. Details and advice can be obtained from the Copyright Manager. Tel: 020 8996 7070.

BSI
389 Chiswick High Road
London
W4 4AL