BRITISH STANDARD

BS 508-2: 1984

CONFIRMED
JUNE 1990

Butan-1-ol for industrial use —

Part 2: Methods of test

Confirmed
January 2011

UDC 661.725.4:543.062

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Chemicals Standards Committee (CIC/-) to Technical Committee CIC/4 upon which the following bodies were represented:

Chemical Industries' Association
Ministry of Defence
Oil and Colour Chemists' Association
Royal Society of Chemistry
Society of Chemical Industry

The following bodies were also represented in the drafting of the standard, through subcommittees and panels:

British Pharmacopoeia Commission
British Society of Perfumers
Cosmetic, Toiletry and Perfumery Association Limited
Department of Trade and Industry (Laboratory of the Government Chemist)

This British Standard, having been prepared under the direction of the Chemicals Standards Committee, was published under the authority of the Board of BSI and comes into effect on
31 October 1984

The following BSI references relate to the work on this standard:
Committee reference CIC/4
Draft for comment 80/51210 DC

ISBN 0 580 14084 9

Amendments issued since publication

Amd. No.	Date of issue	Comments

Content

Foreword

This Part of this British Standard, which has been prepared under the direction of the Chemicals Standards Committee, provides a comprehensive series of test methods for butan-1-ol for industrial use. Although applicable to the material in general, it includes all the test methods required to assess compliance with BS 508-1. The standard previously numbered BS 508 (published in 1966) has been amended to become BS 508-1 "*Specification*" and the methods of test that were included in the appendices have been deleted.

In preparing this standard, the opportunity has been taken to implement Parts 1 and 2 of ISO 755 "*Butan-1-ol for industrial use — Methods of test*", published by the International Organization for Standardization (ISO). Part 3, although approved by the UK, is not to be implemented as there is no UK requirement for the test method. These standards have been prepared, with the active participation of the UK, by Technical Committee 47, Chemistry, of the International Organization for Standardization (ISO), as separate parts of ISO 755 and constitute the revision of ISO Recommendation R 755. Subsequent International Standards in the ISO 755 series, if approved by the UK, will be published as additions to this British Standard, without technical alteration.

Clauses **2** to **9** and clause **11** of this British Standard are related to ISO 755-1 but not equivalent in technical content. The technical differences occur in the method for the determination of aldehydes and ketones content (see clause **9**): in this British Standard only one concentration of potassium hydroxide solution is allowed for the titration and only one form of expression of the results (as a percentage by mass calculated as butyraldehyde) is given. ISO 755-1 refers to the general method described in ISO 1843-3 for the determination of aldehydes and ketones content, and gives the additional information needed to carry out the determination for butan-1-ol: in this British Standard, the method is described in full.

Clause **10** of this standard, which describes a method for the determination of acidity, is equivalent to ISO 755-2 in technical content but differs in presentation.

This Part of this standard describes methods of test only and should not be referred to as a specification defining limits of purity. Reference to this Part of this standard should indicate that the methods of test used are in accordance with the appropriate clause(s) of BS 508-2.

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 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.

1 Scope

This Part of this British Standard describes methods for testing butan-1-ol for industrial use.

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

2 Sampling[1)]

Store the laboratory sample in a clean, dry and airtight, ground glass stoppered bottle, or a screw-capped bottle fitted with a polyethylene cone insert, of such capacity that it is almost filled by the sample. Sufficient ullage should be left in the bottle to avoid excessive pressure changes that could arise from temperature variations during storage and handling. About 10 % ullage is recommended. If it is necessary to seal the bottle, take care to avoid any risk of contamination of the contents. Store the sample in a cool place in the dark.

3 Determination of density at 20 °C

Determine the density at 20 °C by the method described in BS 4522.

4 Determination of distillation characteristics

Determine the distillation characteristics by the method described in BS 4591, except that the following thermometer, temperature corrections and distillation conditions shall be used.

a) *Thermometer* (**3.2** of BS 4591:1971). Use a thermometer designated F150C/100 complying with the requirements of BS 593.

b) *Corrections to be applied to observed temperatures* (**7.2** of BS 4591:1971). If the corrected barometric pressure deviates from 1013 mbar[2)] apply a correction to the observed temperature by subtracting 0.028 °C for every millibar above, or adding 0.028 °C for every millibar below, 1013 mbar.

c) *Distillation* (**6.1** of BS 4591:1971). Regulate the rate of heating so that the first drop of distillate falls from the end of the condenser after 10 to 15 min.

5 Determination of residue on evaporation on a water bath

Determine the residue on evaporation by the method described in BS 4524.

6 Determination of bromine number

Determine the bromine number (bromine index) by the method described in BS 4523.

7 Measurement of colour

Determine the colour by the method described in BS 5339.

8 Determination of water content

Determine the water content by one of the methods described in BS 2511.

9 Determination of aldehydes and ketones content

NOTE This method is based on ISO 1843-3 which is to be implemented as part of the revision of BS 4583:1970.

9.1 Principle

Reaction of carbonyl compounds present in a test portion with hydroxylammonium chloride to form an oxime, and potentiometric titration of the hydrochloric acid liberated with standard volumetric ethanolic potassium hydroxide solution.

9.2 Reagents

9.2.1 *General*. During the analysis, use only reagents of recognized analytical grade and only water complying with BS 3978.

NOTE The ethanol specified in **9.2.2** and **9.2.4** may be replaced for these purposes by industrial methylated spirits, 95 % (*V*/*V*) complying with BS 3591. It should be noted that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations, 1983 (S.I. 1983 No. 252). It is not permissible to use duty-free ethanol, received under the provisions of The Alcoholic Liquor Duties Act 1979, Section 10, for purposes for which industrial methylated spirits is an acceptable alternative to ethanol.

9.2.2 *Hydroxylammonium chloride*, 10 g/L ethanolic solution.

Dissolve 50g of hydroxylammonium chloride ($NH_2OH.HCl$) in 90 mL of water and dilute to 1 000 mL with 95 % (*V*/*V*) ethanol. Further dilute 100 mL of this solution to 500 mL with 95 % (*V*/*V*) ethanol.

WARNING. Hydroxylammonium chloride is toxic, corrosive and an irritant. Avoid contact with eyes and skin.

[1)] Additional guidance is given in BS 5309-1 and BS 5309-3.

[2)] 1 mbar = 100 N/m^2 = 100 Pa.

9.2.3 *Ethanol*, anhydrous, carbonyl-free, prepared as follows.

Boil under reflux 500 mL of anhydrous ethanol with 5 g of 2,4-dinitrophenylhydrazine and 5 drops of hydrochloric acid solution, ρ approximately 1.19 g/mL, for 2 to 3 h. Distil off the ethanol slowly using a Widmer distillation column about 300 mm long and about 25 mm in diameter, or any other suitable column. Reject the first 50 mL of distillate and collect the next 400 mL, rejecting the remainder. If the distillate is found to be coloured, carry out a redistillation.

9.2.4 *Potassium hydroxide*, standard volumetric solution in 95 % (*V*/*V*) ethanol, *c* (KOH) = 0.1 mol/L.

9.3 Apparatus

9.3.1 *Ordinary laboratory apparatus*

9.3.2 *Two conical flasks*, of borosilicate glass, of capacity 250 mL, fitted with ground glass stoppers.

9.3.3 *Two water-cooled reflux condensers*, with ground glass joints to fit the flasks (**9.3.2**).

9.3.4 *pH meter*, fitted with a glass measuring electrode and a calomel reference electrode.

9.4 Procedure

9.4.1 *Test portion*. Into one of the conical flasks (**9.3.2**) already containing 10 mL of the hydroxylammonium chloride solution (**9.2.2**) weigh, to the nearest 0.001 g, 25 g to 30 g of the laboratory sample.

9.4.2 *Blank test*. Carry out a blank test at the same time as the determination, following the same procedure but omitting the test portion.

9.4.3 *Determination*. Add 10 mL of the ethanol (**9.2.3**) to the conical flask (**9.3.2**) containing the test portion (**9.4.1**). Attach one of the condensers (**9.3.3**) to the flask and reflux for 30 min on a boiling water bath. Remove the flask, still carrying its condenser, from the boiling water bath and allow to cool to ambient temperature. When cold, wash down the inside of the condenser with 10 mL of the ethanol. Transfer the contents of the flask quantitatively to a 400 mL beaker, washing with 125 mL of the ethanol.

Titrate with the potassium hydroxide solution (**9.2.4**) using the pH meter (**9.3.4**). The volume/potential graph may be plotted directly, in which case the point of inflection corresponds to the end-point of the titration (pH value about 3). Alternatively, the first derived curve may be plotted, in which case the end-point of the titration corresponds to the turning point of the curve.

9.5 Expression of results

The aldehydes and ketones content, expressed as a percentage by mass of butyraldehyde (C_3H_7CHO), is given by the formula

$$\frac{0.72\ (V_1 - V_0)}{m}$$

where

V_0 is the volume of the potassium hydroxide solution used for the blank test (in mL);

V_1 is the volume of the potassium hydroxide solution used for the determination (in mL);

m is the mass of the test portion (in g).

10 Determination of acidity

10.1 Principle

Titration of the acidity of a test portion with a standard volumetric sodium hydroxide solution, using phenolphthalein as indicator.

10.2 Reagents

10.2.1 *General*. During the analysis, use only reagents of recognized analytical grade and only water complying with BS 3978.

10.2.2 *Sodium hydroxide*, standard volumetric solution, *c* (NaOH) = 0.1 mol/L.

10.2.3 *Phenolphthalein*, 5 g/L ethanolic solution.

Dissolve 0.5 g of phenolphthalein in 100 mL of 95 % (*V*/*V*) ethanol, and add the sodium hydroxide solution (**10.2.2**) until a pale pink colouration is obtained.

NOTE The ethanol may be replaced for this purpose by industrial methylated spirits, 95 % (*V*/*V*) complying with BS 3591. It should be noted that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations, 1983 (S.I. 1983 No. 252). It is not permissible to use duty-free ethanol, received under the provisions of The Alcoholic Liquor Duties Act 1979, Section 10, for purposes for which industrial methylated spirits is an acceptable alternative to ethanol.

10.3 Apparatus

10.3.1 *Ordinary laboratory apparatus*

10.3.2 *Conical flask*, of capacity 250 mL, of borosilicate glass, fitted with a ground glass stopper.

10.3.3 *Burette*, of capacity 10 mL, graduated in 0.02 mL divisions, complying with class A of BS 846.

10.4 Procedure

10.4.1 *Test portion*. Weigh, in the conical flask (**10.3.2**), 100 ± 0.1 g of the laboratory sample.

10.4.2 *Determination*. Add 0.5 mL of the phenolphthalein solution (**10.2.3**) to the flask containing the test portion (**10.4.1**) and titrate with the sodium hydroxide solution (**10.2.2**) until the pink colour remains for 15 s. Shake the contents of the flask, with the stopper in position, after each addition of sodium hydroxide solution.

10.5 Expression of results

The acidity, expressed as a percentage by mass of butyric acid (C_3H_7COOH), is given by the formula

$$0.008\ 8 \times V \times \frac{100}{m}$$

$$= \frac{0.88 \times V}{m}$$

where

V is the volume of the sodium hydroxide solution used for the determination (in mL);

m is the mass of the test portion (in g);

0.008 8 is the mass (in g) of butyric acid corresponding to 1 mL of sodium hydroxide solution, c (NaOH) = 0.100 mol/L.

NOTE If the standard volumetric solution used does not have exactly the concentration stated in the list of reagents, an appropriate correction should be applied.

11 Test reports

The test report, for each determination, should contain the following:

a) an identification of the sample;

b) a reference to the method used;

c) the results, and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in the appropriate clause of this standard or in the British Standards to which reference is made, or regarded as optional.

blank

Publications referred to

BS 593, *Laboratory thermometers.*

BS 846, *Specification for burettes.*

BS 2511, *Methods for the determination of water (Karl Fischer method).*

BS 3591, *Industrial methylated spirits.*

BS 3978, *Water for laboratory use.*

BS 4522, *Method for the determination of density of liquids at 20 °C.*

BS 4523, *Method for the determination of bromine index.*

BS 4524, *Method for determination of residue on evaporation on a water bath.*

BS 4583, *Methods of test for higher alcohols.*

BS 4591, *Method for the determination of distillation characteristics.*

BS 5309, *Methods for sampling chemical products.*

BS 5309-1, *Introduction and general principles.*

BS 5309-3, *Sampling of liquids.*

BS 5339, *Method of measurement of colour in Hazen units (platinum-cobalt scale) of liquid chemical products.*

ISO 755, *Butan-1-ol for industrial use — Methods of test.*

ISO 755-1, *General*[3)].

ISO 755-2, *Determination of acidity — Titrimetric method*[3)].

ISO 755-3, *Sulphuric acid colour test*[3)].

ISO 1843-3, *Higher alcohols for industrial use — Methods of test — Part III: Determination of carbonyl compounds content — Potentiometric method.*

[3)] Referred to in the foreword only.

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

BSI
389 Chiswick High Road
London
W4 4AL