BRITISH STANDARD

BS 1994:1993

Specification for

Dichloromethane (methylene chloride)

Confirmed
January 2011

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Chemicals Standards Policy Committee (CIC/-) to Technical Committee CIC/51, upon which the following bodies were represented:

British Pharmacopoeia Commission
British Society of Perfumers
Chemical Industries' Association
Health and Safety Executive
Imperial Chemical Industries Limited
Institute of Refrigeration
Oil and Colour Chemists' Association
Royal Society of Chemistry
Solvents Industry Association Ltd.

This British Standard, having been prepared under the direction of the Chemicals Standards Policy Committee, was published under the authority of the Standards Board and comes into effect on 15 November 1993

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The following BSI references relate to the work on this standard:
Committee reference CIC/51
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Amendments issued since publication

Amd. No.	Date	Comments

Contents

Foreword

This British Standard, which has been prepared under the direction of the Chemicals Standards Policy Committee, comprises a specification for dichloromethane.

BS 1994 was first published in 1953 as one of a series of standards for solvents and allied products. This new edition of BS 1994 supersedes BS 1994:1953, which is withdrawn. Although the technical content remains the same, changes have been made to use up-to-date terminology and SI units and to eliminate procedures which are considered potentially unsafe (e.g. of asbestos).

This British Standard is related to ISO 1869:1977 but is not equivalent in technical content. ISO 1869 lists various test methods for dichloromethane.

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 British Standard specifies requirements for dichloromethane suitable for industrial purposes.

2 References

2.1 Normative references

This standard incorporates, by reference, provisions from specific editions of other publications. These normative references are cited at the appropriate points in the text and the publications are listed on the inside back cover. Subsequent amendments to, or revisions of, any of these publications apply to this standard only when incorporated in it by updating or revision.

2.2 Informative references

This standard refers to other publications that provide information or guidance. Editions of these publications current at the time of issue of this standard are listed on the inside back cover, but reference should be made to the latest editions.

3 Description

The material shall be clear, colourless and free from matter in suspension and shall consist essentially of dichloromethane, CH_2Cl_2.

4 Sampling and size of sample

A representative sample of the material measuring not less than 1 l shall be taken from the bulk for the purpose of examination in accordance with this standard. The sample shall be placed in a clean, dry and air-tight ground glass-stoppered bottle of dark amber glass of such capacity that it is nearly filled by the sample. The sample shall be kept in the dark.

NOTE 1 Detailed information on the sampling of liquid chemical products is given in BS 5309-1:1976 and BS 5309-3:1976.

NOTE 2 When it is necessary to seal the container, care should be taken to avoid the risk of contaminating the contents in any way.

NOTE 3 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.

5 Density

The density of the material at 20 °C, when determined by the method described in BS 4522:1988, shall be not lower than 1.321 g/ml and not higher than 1.331 g/ml.

6 Distillation range

When the material is distilled by the method described in BS 4591:1990, modified as described in Annex A of this standard, at 101.3 kPa pressure it shall yield not less than 95 % by volume between 39.0 °C and 40.5 °C.

7 Residue on evaporation

The residue on evaporation of the material shall not exceed 0.01 % (*m*/*m*) when determined by the method described in BS 4524:1983.

8 Water content

The material shall contain not more than 0.05 % (*m*/*m*) of water when determined by one of the methods described in clause **5** of BS 2511:1970 using 20 g of the material.

9 Acidity

The acidity of the material, calculated as hydrochloric acid, HCl, shall not exceed 0.001 % (*m*/*m*) when determined by the method described in Annex B.

10 Free chlorine

The material shall not show any free chlorine when determined by the method described in Annex C.

Annex A (normative) Determination of distillation range

Determine the distillation range, as percentage yield by volume between 39.0 °C and 40.5 °C, in accordance with BS 4591:1990 with the following modifications.

a) *Thermometer* (see **5.1.2** of BS 4591:1990). Use a thermometer designated F150C/100 conforming to BS 593:1989.

b) *Distillation* (see **7.2** of BS 4591:1990). Regulate the rate of heating so that the first drop of distillate falls from the end of the condenser after 10 min to 15 min.

c) *Corrections to be applied to observed temperatures* (see **9.2.2** of BS 4591:1990). If the corrected barometric pressure deviates from 101.3 kPa, correct the observed temperatures by subtracting 0.038 °C for every 1.0 kPa above 101.3 kPa, or adding 0.038 °C for every 1.0 kPa below 101.3 kPa.

NOTE These corrections are valid only for pressures above 93.3 kPa.

Annex B (normative) Determination of acidity

B.1 Principle

A test portion is diluted with carbon dioxide free water and titrated with standard volumetric sodium hydroxide solution, using phenolphthalein indicator.

B.2 Reagents

B.2.1 *General*

During the analysis use only reagents of recognized analytical grade, only water conforming to grade 3 of BS 3978:1987 and only methylated spirits conforming to BS 3591:1985.

NOTE The use of industrial methylated spirits is governed by the Methylated Spirits Regulations, 1983 (S.I. 1983 No 252 [1]). It is not permissible to use duty-free ethanol, received under the provisions of the Alcoholic Liquor Duties Act 1979, Section 10 [2], for purposes for which industrial methylated spirits is an acceptable alternative to ethanol.

B.2.2 *Sodium hydroxide,* standard volumetric solution, c(NaOH) = 0.100 mol/l.

B.2.3 *Phenolphthalein*, 5 g/l ethanolic solution, prepared by dissolving 0.5 g of phenolphthalein in 100 ml of 95 % (*V*/*V*) ethanol or 95 % (*V*/*V*) industrial methylated spirits and adding the sodium hydroxide solution (**B.2.2**) until a pale pink colour is obtained.

B.3 Apparatus

B.3.1 *Ordinary laboratory apparatus.*

B.3.2 *Conical flask,* of 500 ml capacity, of borosilicate glass, fitted with a ground glass stopper carrying a guard tube containing sodium hydroxide on an inert support (soda lime).

B.3.3 *Burette,* of 10 ml capacity, graduated in 0.02 ml divisions, conforming to class A of BS 846:1985.

B.4 Procedure

B.4.1 ***Test portion***

Take 100 ml ± 1 ml of the sample, measured at 20 °C.

B.4.2 ***Determination***

Place 100 ml of water and a few clean anti-bumping granules in the conical flask (**B.3.2**) and boil gently for 5 min to remove any carbon dioxide and allow to cool. Add the test portion (**B.4.1**) and shake vigorously. Allow the layers to separate, add 0.5 ml of the phenolphthalein solution (**B.2.3**) and titrate with the sodium hydroxide solution (**B.2.2**), using the burette (**B.3.3**) until a pink colour, persisting for about 15 s, is obtained.

B.5 Expression of results

The acidity A, expressed as a percentage by mass of hydrochloric acid, is given by the equation:

$$A = \frac{0.00365 \times V_1}{\rho}$$

where

V_1 is the volume of sodium hydroxide solution used for the determination (in ml);

ρ is the density of the sample at 20 °C (determined by the method described in BS 4522:1988) (in g/ml);

0.00365 is the mass of hydrochloric acid corresponding to 1.00 ml of sodium hydroxide solution, c(NaOH) = 0.100 mol/l (in g).

Report the value of A as a percentage (m/m) to two significant figures.

Annex C (normative)
Detection of free chlorine

C.1 Principle

The presence of free chlorine is indicated by development of a pink colour with the addition of 3,3'-dimethylnaphthidine solution.

C.2 Reagent

C.2.1 3,3′-*Dimethylnaphthidine solution,* 0.05 g/l, prepared by dissolving 0.01 g of finely ground 3,3′-dimethylnaphthidine in 5 ml of glacial acetic acid in a 200 ml one-mark graduated flask and diluting rapidly with water conforming to grade 3 of BS 3978:1987. Store the solution in the dark.

C.3 Procedure

C.3.1 *Test portion*

Take 50 ml ± 0.5 ml of the sample.

C.3.2 *Detection*

Place the test portion (**C.3.1**) into a glass stoppered 50 ml measuring cylinder, add 5 ml of the 3,3′-dimethylnaphthidine solution (**C.2.1**), stopper and shake the cylinder for 30 s. Note whether a pink colour develops, indicating the presence of free chlorine.

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List of references

Normative references

BSI Standards publications

BRITISH STANDARDS INSTITUTION, London

BS 593:1989, *Specification for laboratory thermometers.*

BS 846:1985, *Specification for burettes.*

BS 2511:1970, *Methods for the determination of water (Karl Fischer method).*

BS 3591:1985, *Specification for industrial methylated spirits.*

BS 3978:1987, *Specification for water for laboratory use.*

BS 4522:1988, *Method for the determination of absolute density at 20 °C of liquid chemical products for industrial use.*

BS 4524:1983, *Method for the determination of residue on evaporation on a water bath.*

BS 4591:1990, *Method for the determination of distillation characteristics of organic liquids (other than petroleum products).*

Informative references

BSI Standards publications

BRITISH STANDARDS INSTITUTION, London

BS 5309, *Methods for sampling chemical products.*

BS 5309-1:1976, *Introduction and general principles.*

BS 5309-3:1976, *Sampling of liquids.*

ISO publications

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION (ISO), Geneva. (All publications are available from Customer Services, Publications, BSI.)

ISO 1869:1977, *Methylene chloride for industrial use — List of methods of test*[1)].

Other references

[1] GREAT BRITAIN. The Methylated Spirits Regulations, 1983 (S.I. No 252). London: HMSO.

[2] GREAT BRITAIN. The Alcoholic Liquor Duties Act 1979, Section 10. London: HMSO.

[1)] Referred to in the foreword only.

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