BRITISH STANDARD

BS 552:1970
Incorporating Amendment No. 1

Specification for

Amyl acetate

Confirmed January 2011

Co-operating organizations

The Chemicals Industry Standards Committee under whose supervision this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

Board of Trade
British Steel Industry
Chemical Industries Association*
Department of Health and Social Security
Fertilizer Manufacturers' Association Ltd
Gas Council
Institution of Gas Engineers
Ministry of Agriculture, Fisheries and Food
Ministry of Technology — Laboratory of the Government Chemist*
National Sulphuric Acid Association
Royal Institute of Public Health & Hygiene

The Government department and industrial organization marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard.

British Plastics Federation
Ministry of Defence, Navy Department
Oil and Colour Chemists' Association
Paintmakers' Association of Great Britain
Pharmaceutical Society of Great Britain
Research Association of British Paint, Colour and Varnish Manufacturers
Royal Institute of Chemistry
Society of Chemical Industry
Society of Motor Manufacturers and Traders Ltd.
Toilet Preparations Federation

This British Standard, having been approved by the Chemicals Industry Standards Committee, was published under the authority of the Executive Board on 23 July 1970

First published May 1934
First revision March 1950
Second revision March 1957
Third revision July 1970

The following BSI references relate to the work on this standard:
Committee references CIC/4, CIC/4/5 and CIC/51
Draft for comment 69/27346

ISBN 580 06071 3

Amendments issued since publication

Amd. No.	Date of issue	Comments
7218	September 1992	Indicated by a sideline in the margin

Contents

Foreword

This standard makes reference to the following British Standards:

BS 593, *Laboratory thermometers.*

BS 612, *Nessler cylinders.*

BS 658, *Apparatus for the determination of distillation range.*

BS 1792, *One-mark volumetric flasks.*

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

BS 3591, *Industrial methylated spirits.*

BS 3978, *Water for laboratory use.*

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

This standard forms one of a series of British Standards for solvents and allied products, the preparation of which was authorized originally by the Fine Chemicals Industry Standards Committee (now merged in the Chemicals Industry Standards Committee).

This British Standard was first issued in 1934, and was last revised in 1957. In the present revision a limit for colour has been introduced.

The opportunity has been taken to clarify the wording of the standard and to bring it into line with current conventions.

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 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 amyl acetate suitable for industrial purposes.

2 Description

British Standard amyl acetate shall be clear and free from matter in suspension, and shall consist essentially of the acetic esters of amyl alcohols, mixtures thereof, or alcohols obtained from fusel oil.

3 Colour

The colour of the material shall not exceed 15 Hazen units when measured by the method described in Appendix A or by a suitable instrumental method.

4 Relative density

The relative density[1)] of the material at any one of the following temperatures shall be within the appropriate values as shown:

Temperature	Relative density	
°C	min.	max.
20/4	0.866	0.874
20/20	0.868	0.876
25/25	0.864	0.872

5 Distillation yield

The material, on distillation by the method described in Appendix B, shall yield at 101.3 kN/m^2[2)] (760 mmHg) pressure not less than 95 % by volume between 120 °C and 145 °C and not more than 67 % by volume below 135 °C.

6 Residue on evaporation

The material shall not leave more than 0.01 % by mass of residue when tested by the method described in Appendix C.

7 Water

The material shall not contain more than 0.3 % by mass of water, determined by the method described in clause **2** of BS 2511:1970[3)] and using 20 ml of the material.

8 Acidity

The material shall not contain more than 0.01 % by mass of acid, calculated as acetic acid, CH_3COOH, and determined by the method described in Appendix D.

9 Ester content

The ester content of the material shall be not less than 95 % by mass, calculated as amyl acetate, $CH_3COOC_5H_{11}$, and determined by the method described in Appendix E.

10 Sampling and size of sample

A representative sample of the material measuring not less than half a litre 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 airtight glass-stoppered bottle of such a size that it is nearly filled by the sample.

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

1) Ratio of the density of a liquid at a specified temperature to the density of water at a specified temperature.

2) 1 kN/m^2 = 10 mbar = 7.5 mmHg.

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

Appendix A Limit test for colour

A.1 Apparatus

The apparatus required comprises:

1) *Matched Nessler cylinders*[4], two of 100 ml capacity.

2) *One-mark volumetric flasks*[5], one of 250 ml capacity and one of 500 ml capacity.

A.2 Reagents

The reagents used shall be of a recognized analytical reagent quality. Water complying with BS 3978[6] shall be used throughout.

1) *Cobaltous chloride*, hexahydrate.

2) *Hydrochloric acid*, concentrated, 36 % m/m (11N).

Either

3) *Chloroplatinic acid reagent*. Dissolve 250 mg of platinum in a small quantity of aqua regia contained in a glass or porcelain basin by heating on a water bath. When the metal has dissolved evaporate the solution to dryness. Add 1 ml of the hydrochloric acid and again evaporate to dryness. Repeat this operation twice more.

or

4) *Potassium chloroplatinate*

A.3 Preparation of colour standard

Dissolve 0.5 g of the cobaltous chloride hexahydrate and either the whole of the chloroplatinic acid (prepared as described above) or 2.490 g of potassium chloroplatinate, in 50 ml of the hydrochloric acid. Warm, if necessary, to obtain a clear solution and after, cooling, pour into the 500 ml volumetric flask. Dilute with water to the mark.

Pipette 7.5 ml of this solution into the 250 ml volumetric flask. Dilute with water to the mark. This diluted solution has a colour of 15 Hazen units and should always be freshly prepared.

A.4 Procedure

Fill one of the Nessler cylinders to the mark with the sample, and the other with the colour standard. Using a white background, compare the colours.

Appendix B Method for the determination of distillation yield

B.1 Apparatus

The apparatus required is described in BS 658[7] and comprises:

1) *Distillation flask* of 150 ml distillation capacity complying with the requirements of BS 658[7].

2) *Thermometer*, No. F255C/100, complying with the requirements of BS 593[8].

3) *Receiver*, Type 1, of 100 ml capacity, complying with the requirements of BS 658[7].

4) *Condenser*, Type 1, complying with the requirements of BS 658[7].

5) *Draught screen*, Type B, complying with the requirements of BS 658[7].

B.2 Corrections to be applied to the specified distillation temperatures before commencing the distillation

B.2.1 Read the barometer and correct the reading as described in clause **8** of BS 4591:1990[9].

B.2.2 When the corrected barometric pressure deviates from 101.3 kN/m^2[10] (760 mmHg), apply corrections to the specified distillation temperatures by adding 0.36 °C for every 1 kN/m^2 above 101.3 kN/m^2 (0.047 °C per millimetre of mercury) or subtracting 0.36 °C for every 1 kN/m^2 below 101.3 kN/m^2 (0.047 °C per millimetre of mercury).

NOTE These corrections are valid only for pressure above 93 kN/m^2 (700 mmHg).

B.2.3 If the thermometer gives incorrect readings at the corrected specified distillation temperatures, further adjust the latter by adding the amount of error if the thermometer is reading high, or subtracting the amount of error if the thermometer is reading low.

---

[4] BS 612, "*Nessler cylinders*".

[5] BS 1792, "*One-mark volumetric flasks*".

[6] BS 3978, "*Water for laboratory use*".

[7] BS 658, "*Apparatus for the determination of distillation range (including flasks and receiver)*".

[8] BS 593, "*Laboratory thermometers*".

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

[10] 1 kN/m^2 = 10 mbar = 7.5 mmHg.

B.3 Procedure

Assemble the apparatus as described in BS 658[7]. Measure 100 ml of the sample in the receiver. Transfer the sample as completely as possible to the distillation flask and add a few anti-bumping granules. Place the flask, thermometer and receiver in position and ensure that the condenser has a steady supply of water. Adjust the rate of heating so that the first drop of distillate falls from the end of the condenser in 10 to 15 minutes. Further adjust the rate of heating so that the distillate is collected at the rate of 3 ml to 4 ml per minute.

Read the volume of distillate in the receiver when the thermometer indicates each of the corrected specified distillation temperatures and express the volumes so recorded as percentages by volume.

Appendix C Method for the determination of residue on evaporation

C.1 Procedure

Pipette 100 ml of the sample into a weighed glass evaporating dish on a boiling water bath. Blow a stream of filtered air onto the surface of the liquid from a jet 1 mm in diameter, with the tip of the jet 10 mm above the edge of the dish and under a pressure of 0.5 kN/m^2 (50 mm of water).

Evaporate the sample to dryness and dry the residue for one hour in an oven, at a temperature of 105 ± 5 °C. Cool in a desiccator and weigh. Dry the residue in the oven again for 30 minutes, cool in a desiccator and weigh again. Repeat, if necessary, until two successive weighings do not differ by more than 1 mg.

C.2 Calculation

Residue on evaporation, per cent by mass

$$= \frac{M}{d}$$

where M = mass, in grammes, of residue

and d = relative density of the sample.

Appendix D Method for the determination of acidity

D.1 Reagents

The reagents used shall be of a recognized analytical reagent quality. Water complying with BS 3978[11] shall be used throughout.

1) *Sodium hydroxide*, 0.1N solution.

2) *Ethanol*[12], 95 % (v/v).

3) *Phenolphthalein indicator solution*, 10 g/l. Dissolve 2.5 g of phenolphthalein in 250 ml of ethanol[12] 95 % (v/v), and make faintly pink by the addition of dilute sodium hydroxide solution.

D.2 Procedure

Take 50 ml of the ethanol, add 0.5 ml of the phenolphthalein indicator and neutralize with sodium hydroxide solution. Add 50 ml of the sample and titrate the mixture immediately with the 0.1**n** sodium hydroxide solution until the first pink colour persists for at least 10 seconds.

D.3 Calculation

Acidity, calculated as acetic acid, CH_3COOH, per cent by mass

$$= \frac{0.012 \times V_1}{d}$$

where V_1 = volume, in millilitres, of 0.1N sodium hydroxide used

and d = relative density of the sample.

[11] BS 3978, "*Water for laboratory use*".

[12] Ethanol may be replaced by industrial methylated spirits, 95 % (v/v), complying with the requirements of 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.

Appendix E Method for the determination of ester content

E.1 Reagents

The reagents used shall be of a recognized analytical reagent quality. Water complying with BS 3978[13] shall be used throughout, and shall be freshly boiled and cooled.

1) *Potassium hydroxide*, approximately N solution in 95 % (v/v) ethanol[14].

2) *Hydrochloric acid*, N solution.

3) *Phenolphthalein indicator solution*, 10 g/l. Dissolve 2.5 g of phenolphthalein in 250 ml of ethanol[14], 95 % (v/v) and make faintly pink by the addition of dilute sodium hydroxide solution.

E.2 Procedure

Measure 50.0 ml of the potassium hydroxide solution into each of two dry 250 ml conical flasks with ground glass jointed stoppers. Close the flasks with their glass stoppers. Transfer by means of a weighing pipette between 3.0 g and 3.5 g of the sample (weighed to the nearest 0.001 g) into one of the flasks.

Attach the flasks to water-cooled reflux condensers with ground glass joints, and heat for one hour in a boiling-water bath. Withdraw the flasks, still carrying their condensers, and immerse them in cold running water. When cool, wash down the inside of each condenser with two 20 ml portions of water. Disconnect the flasks and wash each joint with a further 20 ml of the water. Add 0.5 ml of phenolphthalein indicator and titrate the mixture immediately with the N hydrochloric acid solution until the pink colour is just discharged.

E.3 Calculation

Ester content, calculated as amyl acetate, $CH_3COOC_5H_{11}$,

$$\text{per cent by mass} = \frac{13.02(V_2 - V_3)}{M_2}$$

where V_2 = volume, in millilitres, of N hydrochloric acid solution required by blank,

V_3 = volume, in millilitres, of N hydrochloric acid solution required by sample

and M_2 = mass, in grammes, of sample taken.

[13] BS 3978, "*Water for laboratory use*".

[14] Ethanol may be replaced by industrial methylated spirits, 95 % (v/v), complying with the requirements of 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.

blank

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