BRITISH STANDARD

BS 579:1957

Specification for

Diethyl ether (technical)

Confirmed January 2011

Co-operating organizations

The Fine and Heavy 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:

Association of British Chemical Manufacturers*
Board of Trade
British Iron and Steel Federation
British Railways, The British Transport Commission
Fertilizer Manufacturers' Association
Gas Council
Institution of Gas Engineers
Ministry of Health
Ministry of Supply*
National Sulphuric Acid Association

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

Admiralty
Association of Cellulose Lacquer Manufacturers
British Plastics Federation
Department of the Government Chemist
Institute of Petroleum
National Paint Federation
Oil and Colour Chemists' Association
Pharmaceutical Society of Great Britain
Royal Institute of Chemistry
Society for Analytical Chemistry
Society of Chemical Industry
Society of Motor Manufacturers and Traders Ltd.
Individual manufacturers

This British Standard, having been approved by the Fine and Heavy Chemicals Industry Standards Committee and endorsed by the chairman of the Chemical Divisional Council, was published by the authority of the General Council on 31 December 1957

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The following BSI references relate to the work on this standard:
Committee reference FHC/4 and FHC/4/5
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Amendments issued since publication

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Contents

Foreword

This standard makes reference to the following British Standards:

BS 571, *Flasks for the determination of distillation range.*

BS 593, *Laboratory thermometers.*

BS 604, *Graduated measuring cylinders.*

BS 605, *Distillation receivers (including Crow receivers).*

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

BS 2511, *Determination of water by the Karl Fischer method.*

This standard forms one of a series of British Standards for solvents and allied products. First issued in 1934, it was revised in 1951 and the present edition has been prepared as the result of a review of the standard in the light of present-day requirements.

The standard does not apply to material intended for medicinal use, which is included in the British Pharmacopœia.

The technical content of the standard is substantially unchanged, but specific gravity limits at 25/25 °C have been introduced for use in tropical climates and a limit for water, determined by the Karl Fischer method, has been set.

The opportunity has also 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 5 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 Description

British Standard diethyl ether (technical) shall be clear, colourless and free from matter in suspension, and shall consist essentially of diethyl ether,[1] $C_2H_5OC_2H_5$.

2 Specific gravity

The specific gravity of the material at any one of the following temperatures shall be within the appropriate values as shown:

Temperature	Specific gravity	
	min.	max.
15.5/15.5 °C	0.719	0.725
20/20 °C	0.714	0.720
25/25 °C	0.709	0.715

3 Distillation range[2]

The material, on distillation in the manner described in Appendix A, shall yield at 760 mmHg pressure not less than 95.0 per cent by volume below 36.0 °C.

4 Water

The material shall not contain more than 1.0 per cent by weight of water, determined in the manner described in BS 2511[3].

5 Residue on evaporation[2]

The material shall not leave more than 0.005 per cent by weight of residue when tested in the manner described in Appendix B.

6 Acidity

The material shall not contain more than 0.002 per cent by weight of acid, calculated as sulphuric acid, H_2SO_4, and determined in the manner described in Appendix C.

7 Peroxides[2]

The material shall not show any peroxide when tested in the manner described in Appendix D.

8 Sulphur compounds

The material shall not show any sulphur compounds when tested in the manner described in Appendix E.

9 Sample

For the purpose of examination under this specification, a representative sample of the material measuring not less than half a litre shall be taken from an original and previously unopened container or from the bulk during packing, as may be arranged. The sample shall be placed in a clean, dry and air-tight glass-stoppered bottle of dark amber glass of such 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] This name is approved by the International Union of Pure and Applied Chemistry.
[2] Owing to the inflammable nature of diethyl ether and its vapour, special care should be taken in performing the tests for distillation range and residue on evaporation. In rare cases, particularly where the material has been improperly stored, there may be an element of risk of explosion in carrying out the distillation and residue on evaporation tests, due to the presence of peroxides. It should always be ensured that peroxides are absent (see Appendix D) before commencing these determinations.
[3] BS 2511, "*Determination of water by the Karl Fischer method*".

Appendix A Method for the determination of distillation range

NOTE Owing to the inflammable nature of diethyl ether and its vapour, special care should be taken in carrying out this determination. In rare cases, particularly where the material has been improperly stored, there may be an element of risk of explosion in carrying out this test, due to the presence of peroxide. It should always be ensured that peroxides are absent (see Appendix D) before commencing this determination.

Apparatus

The apparatus required, part of which is shown in Figure 1, comprises:

a) *Distillation flask* of 100 ml distillation capacity, complying with BS 571[4)].

b) *Thermometer* No. F50C/100, complying with BS 593[5)].

c) *Draught screen,* complying with BS 658[6)], except that the shelf shall have a central circular hole 60 mm in diameter, the edge of which is bevelled to fit the contours of the distillation flask.

d) *Water bath* with a steam coil, electrical immersion heater or other suitable heating device for maintaining the temperature of the water, which is determined by means of a thermometer No. C100C/50 complying with BS 593, fitted into that portion of the shelf which forms the lid of the water bath.

e) *Receiver,* Type 1, of 100 ml capacity, complying with BS 605[7)], closed with a plug of cotton wool.

f) *Condenser,* Type 3, complying with BS 658, equipped with a suitable thermometer.

Corrections to be applied to the specified distillation temperatures before commencing the distillation

1) Read the barometer and apply the corrections as described in BS 658.

2) When the corrected barometric pressure, p, deviates from 760 mmHg, add the correction $0.037\ (p - 760)$ Centigrade degrees to the specified distillation temperatures.

NOTE This correction is valid only for pressures above 700 mmHg.

3) If the thermometer gives incorrect readings at the corrected specified distillation temperatures, further correct 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.

Procedure

Assemble the apparatus as described in BS 658, filling the condenser cooling bath with water at a temperature of 5 ± 2 °C and immersing the receiver up to the 90 ml mark in a bath of water maintained at the same temperature. Place the water bath on its support so that it is flush with the underside of the shelf of the draught screen and fill it to within ⅛ in. of the top with water at 60 °C. Measure in the receiver 100 ml of the diethyl ether previously brought to the temperature of the condenser water, transfer as completely as possible to the distillation flask and add a few pieces of clean, dry porous pot. Place the flask, thermometers and receiver in position, close the neck of the receiver with a plug of cotton wool and ensure, by addition of ice, that the condenser cooling bath remains at a temperature of 5 ± 2 °C. Regulate the supply of heat to the water bath so that the temperature of the water is maintained at 60 ± 2 °C throughout the distillation. This should ensure a distillation rate of 3 to 4 ml per minute.

Read the volume of distillate in the receiver when the flask thermometer just reaches either, *a.* the corrected specified distillation temperature or, *b.* the maximum temperature of the distillation, whichever is the lower.

[4)] BS 571, "*Flasks for the determination of distillation range*".
[5)] BS 593, "*Laboratory thermometers*".
[6)] BS 658, "*Apparatus for the determination of distillation range*".
[7)] BS 605, "*Distillation receivers (including Crow receivers)*".

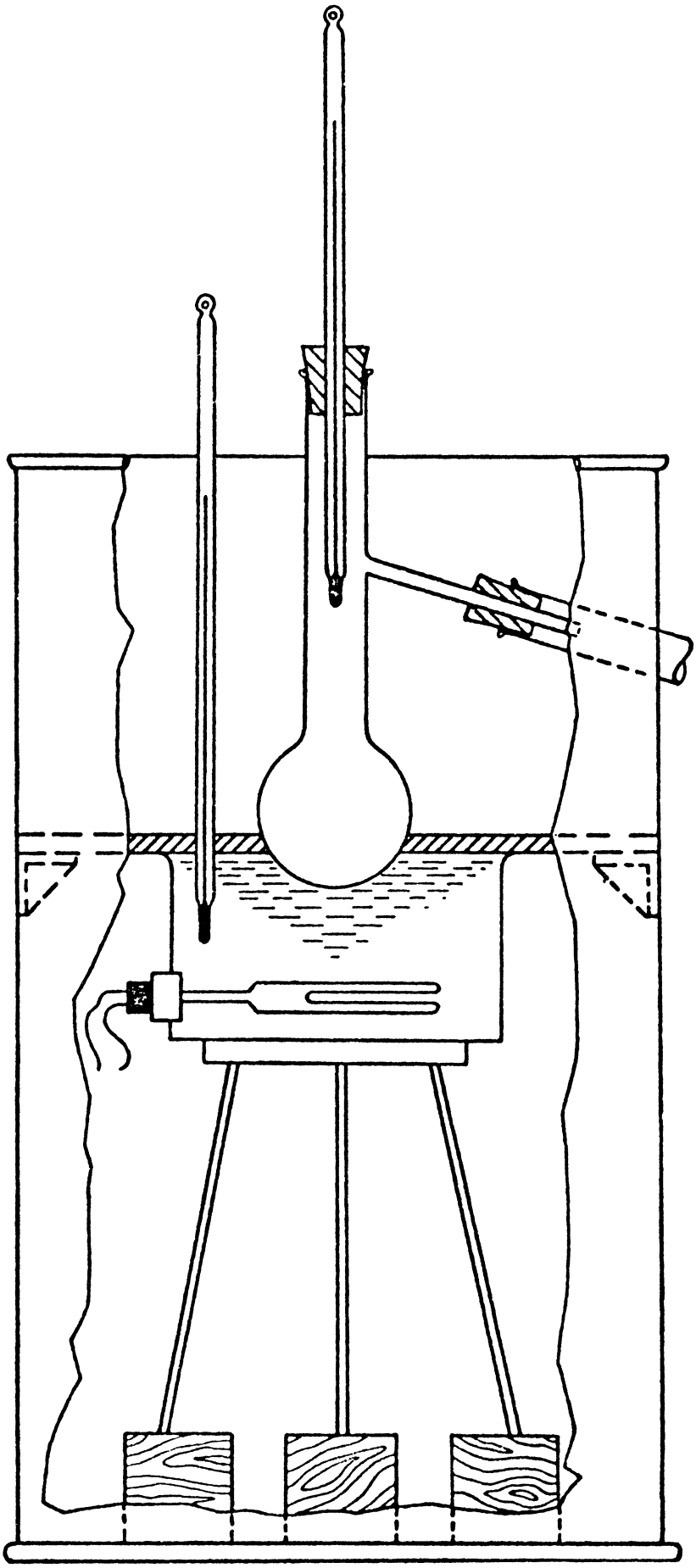

Figure 1 — Assembly of distillation flask, draught screen and water bath

Appendix B Method for the determination of residue on evaporation

NOTE Owing to the inflammable nature of diethyl ether and its vapour, special care should be taken in carrying out this determination. In rare cases, particularly where the material has been improperly stored, there may be an element of risk of explosion in carrying out this test, due to the presence of peroxides. It should always be ensured that peroxides are absent (see Appendix D) before commencing this determination.

Procedure

Evaporate 100 ml of the diethyl ether to dryness in a weighed platinum, silica or borosilicate glass basin on a water bath. Dry the residue for 30 minutes in an oven at a temperature of 100 ± 2 °C. Cool in a desiccator and weigh.

Calculation

$$\text{Residue on evaporation, per cent by weight} = \frac{W}{S}$$

where W = weight in grammes of residue

and S = specific gravity of the diethyl ether.

Appendix C Method for the determination of acidity

Reagents

The reagents used shall be of recognized analytical reagent quality. The distilled water used shall be freshly boiled and cooled.

a) *Sodium hydroxide,* 0.1 N solution.

b) *Hydrochloric acid,* 0.1 N solution.

c) *Methyl red indicator.* Dissolve 0.5 g of water-soluble methyl red in 1 000 ml of distilled water.

Procedure

Transfer 50 ml of the diethyl ether to a glass-stoppered separating funnel, add 50 ml of the distilled water and shake the liquids vigorously. After separation run the aqueous layer into a receiver provided with a stopper. Repeat the shaking out with water twice, each time running the aqueous layer into the receiver already used.

To the combined aqueous layers add 0.2 ml of the methyl red indicator and titrate with the sodium hydroxide solution, using a micro burette.

Perform a blank determination on 150 ml of the distilled water used, using the hydrochloric acid or the sodium hydroxide solution as appropriate. (Distilled water is usually alkaline to methyl red.)

Calculation

Acidity, expressed as sulphuric acid,

$$H_2SO_4\text{, per cent by weight} = \frac{0.0098\ (T_1 + T_2 - T_3)}{S}$$

where T_1 = volume in millilitres of 0.1 N sodium hydroxide solution used for test,

T_2 = volume in millilitres of 0.1 N hydrochloric acid used for blank,

or T_3 = volume in millilitres of 0.1 N sodium hydroxide solution used for blank

and S = specific gravity of the diethyl ether.

Appendix D Method for the detection of peroxides

Apparatus

Stoppered graduated cylinder, of 10 ml capacity, complying with BS 604.[8)]

Reagent

The reagent used shall be of recognized analytical reagent quality.

Potassium iodide, 10 per cent solution, freshly prepared.

[8)] BS 604, "*Graduated measuring cylinders*".

Procedure

Transfer 5 ml of the diethyl ether to the cylinder and add the potassium iodide solution until the ether overflows. Shake vigorously and set aside in the dark for 30 minutes.

Note if a yellow or brown coloration, indicating the presence of peroxides, develops in either layer.

Appendix E Method for the detection of sulphur compounds

Reagents

The reagents used shall be of recognized analytical reagent quality.

a) *Acetic acid,* 10 per cent.

b) *Mercury,* clean, redistilled.

Procedure

To 10 ml of the diethyl ether contained in a glass-stoppered cylinder add 1 ml of the acetic acid and approximately 0.2 ml of the mercury. Shake vigorously for one minute and then allow to stand for one hour.

Note if a dark brown or black precipitate, indicating the presence of sulphur compounds, settles out at the ether-water interface.

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