BRITISH STANDARD

BS 2068:1970

Incorporating Amendment No. 1

Specification for

Acetic anhydride

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 industrial organizations:

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British Steel Industry
Chemical Industries Association*
Department of Health and Social Security
Fertilizer Manufacturers' Association
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 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.

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Ministry of Technology — Laboratory of the Government Chemist
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Paintmakers Association of Great Britain
Pharmaceutical Society of Great Britain
Research Association of British Paint, Colour & Varnish Manufacturers
Royal Institute of Chemistry
Society of Chemical Industry
Society of Motor Manufacturers and Traders Ltd.

This British Standard, having been approved by the Chemicals Industry Standards Committee, was published under the authority of the Executive Board on 20 January 1970

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Amendments issued since publication

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7627	May 1993	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 846, *Burettes and bulb burettes.*

BS 1792, *One-mark volumetric flasks.*

BS 3591, *Industrial methylated spirits.*

BS 3978, *Water for laboratory use.*

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

This British 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 standard was first issued in 1953. In the present revision the limits for ash, chlorides, sulphates and anhydride content have been made more stringent. The requirements in respect of distillation range have been made more stringent, and now specify initial boiling point and dry point. A specific colour limit has been included. The methods of test specified for distillation range and ash are based on those in ISO Recommendation R 754[1]. In the limit tests for inorganic chlorides and sulphates the mass of sample taken is less than in the ISO methods, because of the difficulty in dissolving so large a mass of sample, and the whole of the test portion is used for the turbidimetric comparison. In the method in Appendix F for determination of anhydride content, cyclohexane is used as solvent in place of benzene which is used in the ISO method and the quantities of methanol used have been adjusted accordingly.

NOTE Attention is drawn to certification facilities offered by BSI; see the back cover of this standard.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 6 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.

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[1] ISO/R754, *"Acetic anhydride for industrial use. Methods of test."*

1 Scope

This British Standard specifies requirements for acetic anhydride suitable for industrial purposes.

2 Description

British Standard acetic anhydride shall be clear and free from matter in suspension, and shall consist essentially of acetic anhydride, $(CH_3CO)_2O$.

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 Distillation range

When the material is distilled by the method described in Appendix B, the initial boiling point at 1 013 mbar[2] (760 mmHg) pressure shall not be below 138.0 °C and the dry point at 1 013 mbar (760 mmHg) pressure shall not be above 141.5 °C.

5 Ash

The material shall not leave more than 100 parts per million by mass of ash when tested by the method described in Appendix C.

6 Inorganic chlorides

The material shall not contain more than 50 parts per million by mass of inorganic chlorides, expressed as Cl and determined by the method described in Appendix D.

7 Inorganic sulphates

The material shall not contain more than 50 parts per million by mass of inorganic sulphates, expressed as SO_4 and determined by the method described in Appendix E.

8 Anhydride content

The material shall show an anhydride content of not less than 98.0 % by mass, calculated as acetic anhydride, $(CH_3CO)_2O$, and determined by the method described in Appendix F.

9 Sample

A representative sample of the material measuring at least 1 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 glass bottle of such a size that it is nearly filled by the sample.

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[2] 1 mbar = 100 N/m^2 = 0.750 mmHg.

NOTE Because of the toxic nature of acetic anhydride the tests described in the appendices should always be carried out under conditions of good ventilation.

Appendix A Limit test for colour

A.1 Apparatus

1) *Two matched Nessler cylinders*[3], 100 ml capacity.

2) *One-mark volumetric flask*[4], 500 ml capacity.

3) *One-mark volumetric flask*[4], 250 ml capacity.

A.2 Reagents

The reagents used shall be of a recognized analytical reagent quality. Water complying with BS 3978[5] shall be used throughout.

1) *Cobaltous chloride*, hexahydrate.

2) *Hydrochloric acid*, concentrated, 36,5 % (m/m) (11N).

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.

A.3 Preparation of colour standard

Dissolve 0.50 g of the cobaltous chloride hexahydrate and the whole of the chloroplatinic acid (prepared as described above) 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 range

B.1 Apparatus

The apparatus required is described in BS 658[6] and comprises:

1) *Distillation flask*, of 130 ml distillation capacity, complying with the requirements of BS 658[6].

2) *Thermometer*, No. F150C/100 complying with the requirements of BS 593[7].

3) *Condenser*, Type 1, complying with the requirements of BS 658[8].

4) *Draught screen*, type C, and *non-asbestos heat-resistant flask support sheet* with 50 mm diameter central hole, complying with the requirements of BS 658[6].

B.2 Procedure

Assemble the apparatus as described in BS 658[6]. Measure 100 ml of the sample into the distillation flask from a graduated measuring cylinder and add a few small anti-bumping granules. Place the flask, thermometer and a suitable 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 12 minutes to 17 minutes. Read the temperature at the instant the first drop falls from the end of the condenser and record as the observed initial boiling point.

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 temperature indicated at the instant the last drop of liquid evaporates from the lowest point in the distillation flask and record as the observed dry point. Disregard any liquid on the side of the flask.

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

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

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

[6] BS 658, "*Apparatus for the determination of distillation range*".

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

[8] BS 658, "*Apparatus for the determination of distillation range*".

B.3 Corrections to be applied to the observed temperatures

1) If the thermometer gives incorrect readings at the observed initial boiling point or observed dry point, correct the readings by subtracting the amount of error if the thermometer is reading high, or adding the amount of error if the thermometer is reading low.

2) Read the barometer and correct the reading as described in BS 4591[9].

3) When the corrected barometric pressure deviates from 1 013 mbar[10] (760 mmHg), apply further corrections to the observed temperatures by subtracting 0.037 °C for every millibar above 1 013 mbar (0.050 °C per millimetre of mercury) or adding 0.037 °C for every millibar below 1 013 mbar (0.050 °C per millimetre of mercury).

NOTE These last corrections are valid only for pressures above 930 mbar (700 mmHg).

Appendix C Method for the determination of ash

C.1 Outline of method

The mass of residue, if any, is determined after evaporation of the sample to dryness and ignition until no carbonaceous matter remains.

C.2 Procedure

Evaporate to dryness in small portions 100 g of the sample in a weighed platinum or silica dish of capacity 50 ml on a steam bath or electric hotplate in a fume cupboard, and gently ignite the residue until no carbonaceous matter remains.

Cool the dish and contents to atmospheric temperature in a desiccator containing anhydrous calcium chloride, and weigh to the nearest 0.2 mg.

C.3 Calculation

$$\text{Ash, parts per million by mass} = \frac{10^6 \times M_1}{M_2}$$

where M_1 = mass, in grammes, of the residue

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

Appendix D Limit test for inorganic chlorides

D.1 Outline of method

The turbidity produced when silver nitrate is added to a solution prepared from the sample in presence of nitric acid is compared with that similarly obtained from a standard chloride solution.

D.2 Apparatus

1) *Nessler cylinders*, 100 ml capacity, complying with BS 612[11].

2) *One-mark volumetric flask*[12], 250 ml capacity.

D.3 Reagents

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

1) *Nitric acid*, approximately 5N solution.

2) *Standard chloride solution*, 1 ml = 0.0001 g Cl. Dilute 28.2 ml of 0.1N hydrochloric acid solution to 1 000 ml with water.

3) *Silver nitrate*, 50 g/l solution.

D.4 Procedure

D.4.1 Weigh 2 g of the sample, and allow it to dissolve in 95 ml of water, cooling if necessary.

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

[10] 1 mbar = 100 N/m² = 0.750 mmHg.

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

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

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

D.4.2 If the solution is not clear, pass it through a chloride-free filter paper. This should remove turbidity due to aluminium.

D.4.3 Transfer the solution prepared from the test sample (see **D.4.1**) to a 100 ml Nessler cylinder, add 2 ml of the nitric acid solution, dilute to the mark with water and mix.

D.4.4 To prepare the chloride solution of known concentration add to a 100 ml Nessler cylinder 1.0 ml of the standard chloride solution, dilute to about 50 ml with water, add 2 ml of the nitric acid solution, dilute to the mark with water and mix. This diluted solution corresponds to a chloride content, expressed as Cl, of 50 parts per million in the original material.

D.4.5 Add to each Nessler cylinder 1 ml of the silver nitrate solution and mix. Allow the cylinders to stand in the dark for 5 minutes then compare the turbidities produced.

Appendix E Limit test for inorganic sulphates

E.1 Outline of method

The turbidity produced when barium chloride is added to a solution prepared from the sample in presence of hydrochloric acid is compared with that similarly obtained from a standard sulphate solution.

E.2 Apparatus

1) *Nessler cylinders*, 100 ml capacity, complying with BS 612[14].
2) *One-mark volumetric flask*[15], 100 ml capacity.

E.3 Reagents

The reagents used shall be of a recognized analytical reagent quality. Water complying with BS 3978[16] shall be used throughout.

1) *Sodium carbonate*, approximately N solution.
2) *Hydrochloric acid*, approximately N solution.
3) *Barium chloride*, $BaCl_2.2H_2O$, 100 g/l solution.
4) *Standard sulphate solution*, 1 ml = 0.0001 g SO_4. Dilute 20.8 ml of 0.1N sulphuric acid solution to 1 000 ml.

E.4 Procedure

E.4.1 Weigh 8 g of the sample into an evaporating basin and allow it to dissolve in about 100 ml of water, cooling if necessary; add 0.2 ml of the sodium carbonate solution and evaporate to dryness on a boiling water bath. Dissolve the residue in 95 ml of water containing 1 ml of the hydrochloric acid solution.

E.4.2 If the solution is not clear, pass it through a sulphate-free filter paper. This should remove turbidity due to aluminium.

E.4.3 Transfer the solution prepared from the test sample (see **E.4.1**) to a 100 ml Nessler cylinder, add 2 ml of the hydrochloric acid solution, dilute to the mark with water and mix.

E.4.4 To prepare the sulphate solution of known concentration, add to a 100 ml Nessler cylinder 4.0 ml of the standard sulphate solution, dilute to about 50 ml with water, add 2 ml of the hydrochloric acid solution, dilute to the mark with water and mix. This diluted solution corresponds to a sulphate content, expressed as SO_4, of 50 parts per million in the original material.

E.4.5 Add to each Nessler cylinder 2 ml of the barium chloride solution and mix. Allow the cylinders to stand for 5 minutes, mix again, and compare the turbidities produced.

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[14] BS 612, "*Nessler cylinders*".
[15] BS 1792, "*One-mark volumetric flasks*".
[16] BS 3978, "*Water for laboratory use*".

Appendix F Method for the determination of anhydride content

F.1 Outline of method

A known amount of the sample is hydrolysed by means of standard sodium hydroxide solution and the alkali consumed is determined by back titration of the excess with standard hydrochloric acid solution.

The same quantity of sample is reacted with aniline in accordance with the equation:

$$(CH_3CO)_2O + C_6H_5NH_2 \longrightarrow C_6H_5NHCOCH_3 + CH_3COOH$$

and the acetic acid formed is titrated using standard sodium hydroxide solution.

The acetic anhydride content is calculated from the difference between the volumes of sodium hydroxide solution consumed respectively in the hydrolysis of the sample and in the titration of the acetic acid formed in its reaction with aniline.

F.2 Apparatus

Burette, Class A, 50 ml capacity, complying with BS 846[17].

F.3 Reagents

The reagents used shall be of a recognized analytical reagent quality. Water complying with BS 3978[18] shall be used throughout.

1) *Sodium hydroxide*, N solution.
2) *Hydrochloric acid*, N solution.
3) *Cyclohexane*, dry, freshly distilled.
4) *Aniline*, dry, freshly distilled.
5) *Methanol*.
6) *Phenolphthalein*. Dissolve 0.5 g of phenolphthalein in 100 ml of 95 % (v/v) ethanol[19] and make it faintly pink by the addition of dilute sodium hydroxide solution.

F.4 Procedure

F.4.1 Dissolve about 2 g of the sample, accurately weighed by means of a weighing pipette, in 50.0 ml of the sodium hydroxide solution contained in a 500 ml flask fitted with a ground glass stopper and allow to stand for 1 hour. Add 40 ml of the cyclohexane, 10 ml of the aniline and 100 ml of the methanol, and titrate the excess of alkali with the hydrochloric acid solution, using 0.5 ml of the phenolphthalein as indicator.

F.4.2 Dissolve about 2 g of the sample, accurately weighed by means of a weighing pipette, in 20 ml of the cyclohexane in a 500 ml flask fitted with a ground glass stopper, cool in ice and add a cold solution of 10 ml of the aniline in 20 ml of the cyclohexane. Allow the mixture to stand for 1 hour in ice.

Add 100 ml of methanol and 50.0 ml of the sodium hydroxide solution. Titrate the excess of alkali with the hydrochloric acid solution, using 0.5 ml of phenolphthalein as indicator.

F.4.3 Carry out a blank test exactly as in **F.4.2** but omitting the sample.

It is essential to take into account corrections arising from calibration of the burette and to correct the volumes of the standard solutions used, for any deviation of temperature from that at which the solutions were standardized.

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[17] BS 846, "*Burettes and bulb burettes*".

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

[19] 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.

F.5 Calculation

Anhydride content, expressed as acetic anhydride, $(CH_3CO)_2O$, per cent by

$$\text{mass} = 10.21 \times \left[\frac{(V_1 - V_2)}{M_5} - \frac{(V_1 - V_3)}{M_6}\right]$$

where V_1 = volume, in millilitres, of N hydrochloric acid solution used in **F.4.3**,

V_2 = volume, in millilitres, of N hydrochloric acid solution used in **F.4.1**,

V_3 = volume, in millilitres, of N hydrochloric acid solution used in **F.4.2**,

M_5 = mass, in grammes, of sample taken in **F.4.1**

and M_6 = mass, in grammes, of sample taken in **F.4.2**.

blank

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