BRITISH STANDARD

BS 549:1970

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Specification for

Diacetone alcohol

Confirmed
January 2011

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Ministry of Agriculture, Fisheries and Food
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This British Standard, having been approved by the Chemicals Industry Standards Committee, was published under the authority of the Executive Board on
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Committee references CIC/4 and CIC/4/5
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Amendments issued since publication

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Foreword

This standard makes reference to the following British Standards:

BS 612, *Nessler cylinders.*

BS 1792, *One-mark volumetric flasks.*

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

BS 2839, *Method for the determination of closed flash-point of petroleum products by means of the Pensky-Martens apparatus.*

BS 3591, *Industrial methylated spirits.*

BS 3978, *Water for laboratory use.*

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 1964. In the present revision the limits for relative density, water content and acidity have been made more stringent. The requirement in respect of miscibility with water has been deleted but a limit for colour has been introduced.

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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.

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1 Scope

This British Standard specifies requirements for diacetone alcohol suitable for industrial purposes.

2 Description

British Standard diacetone alcohol shall be clear and free from matter in suspension, and shall consist essentially of 4-hydroxy-4-methylpentan-2-one, $CH_3COCH_2C(OH)(CH_3)_2$.

3 Colour

The colour of the material shall not exceed 25 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	0.936	0.939
20/20	0.938	0.941
25/25	0.934	0.937

5 Refractive index

The refractive index of the material at 20 °C for the sodium D line shall be not lower than 1.4230 and not higher than 1.4245.

6 Flash point

The flash point of the material, determined by the method described in BS 2839[2)] shall be not lower than 52.0 °C.

7 Water

The material shall not contain more than 0.25 % by mass of water, determined by the method described in BS 2511[3)], Part 4 and using 20 ml of the material.

8 Residue on evaporation

The material shall not leave more than 0.02 % by mass of residue when tested by the method described in Appendix B.

9 Acidity

The material shall not contain more than 0.010 % by mass of acid, calculated as acetic acid, CH_3COOH, and determined by the method described in Appendix C.

10 Sampling and size of sample

A representative sample of the material measuring not less than one litre shall be taken from the bulk for the purpose of examination in accordance with this specification. 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) BS 2839, "*Method for the determination of closed flash-point of petroleum products by means of the Pensky-Martens apparatus*".
3) BS 2511, "*Methods for the determination of water (Karl Fischer method)*".

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Appendix A Limit test for colour

A.1 Apparatus

The following apparatus is required:

1) *Two matched Nessler cylinders*[4], 100 ml. capacity.

2) *One-mark volumetric flask*[5], 500 ml capacity.

3) *One-mark volumetric flask*[5], 250 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 0.50 g of the cobaltous chloride hexahydrate and either the whole of the chloroplatinic acid (prepared as described above) or 0.623 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 and mix well. This solution has a colour of 500 Hazen units.

Pipette 12.5 ml of this solution into the 250 ml volumetric flask. Dilute with water to the mark. This diluted solution has a colour of 25 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 residue on evaporation

B.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 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 the desiccator and weigh again. Repeat, if necessary, until two successive weighings do not differ by more than 1 mg.

B.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 C Method for the determination of acidity

C.1 Reagents

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

1) *Sodium hydroxide*, 0.1N standard volumetric solution.

2) *Phenolphthalein indicator solution*, 10 g/l. Dissolve 2.5 g of phenolphthalein in 250 ml of ethanol[7], 95 % (v/v) and make faintly pink by the addition of dilute sodium hydroxide solution.

[4] BS 612, "*Nessler cylinders*".
[5] BS 1792, "*One-mark volumetric flasks*".
[6] BS 3978, "*Water for laboratory use*".
[7] Ethanol may be replaced by industrial methylated spirits, 66 degrees O.P., complying with BS 3591. It should be noted that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations 1952 (S.I. 1952, No. 2230).

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C.2 Procedure

Place 100 ml of water and a few pieces of clean porous pot in a 500 ml conical flask of borosilicate glass and boil gently for five minutes to eliminate carbon dioxide. Cool slightly and add 50 ml of the sample. Boil gently for a further 5 minutes. At the end of this period close the neck of the flask with a stopper carrying a soda-lime tube and allow to cool. When cold remove the stopper, add 0.5 ml of the plenolphthalein indicator and titrate with the sodium hydroxide solution using a microburette.

C.3 Calculation

Acidity, calculated as acetic acid, CH_3COOH, per cent

$$\text{by mass} = \frac{0.012 \times V}{d}$$

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

and d = relative density of the sample.

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