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Specification for

# Butanone (ethyl methyl ketone) for industrial use

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# Committees responsible for this British Standard

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

British Pharmacopoeia Commission

British Society of Perfumers

Chemical Industries Association

Department of Trade and Industry (Laboratory of the Government Chemist)

Royal Society of Chemistry

This British Standard, having been prepared under the direction of the Chemicals Standards Committee, was published under the authority of the Board of BSI and comes into effect on 28 August 1987

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The following BSI references relate to the work on this standard:

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# Contents

		Page
Cor	nmittees responsible	Inside front cover
Foreword		ii
1	Scope	1
2	Description	1
3	Sampling and size of sample	1
4	Colour	1
5	Density	1
6	Distillation range	1
7	Residue on evaporation	1
8	Water content	1
9	Acidity	1
10	Alcoholic impurities	1
App	pendix A Determination of distillation range	2
App	pendix B Determination of acidity	2
App	pendix C Determination of alcoholic impurities	2
Pul	plications referred to	Inside back cover

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## **Foreword**

This British Standard, which has been prepared under the direction of the Chemicals Standards Committee, comprises a specification for butanone to meet the requirements of a wide range of industrial users.

This British Standard supersedes BS 1940:1968 which is withdrawn. In this revision of BS 1940 the requirement for water content has been made more stringent and the requirement for relative density has been replaced by one for density at 20  $^{\circ}$ C.

This British Standard is related to ISO 2497:1973 but is not equivalent in technical content. ISO 2497 is published by the International Organization for Standardization (ISO). ISO 2497 lists various general test methods and test methods specifically for butanone, and many of these methods correspond to those which are referred to in this British Standard; ISO 2497 does not specify limits for any of the properties of butanone.

Appendix B of this British Standard is related to ISO 2887:1973 but is not equivalent in technical content. ISO 2887 is published by ISO. The main differences are that in the method described in ISO 2887 the test portion is diluted with either ethanol or propan-2-ol and carbon dioxide is removed by passing a stream of nitrogen through the solution.

Appendix C of this British Standard is related to ISO 2501:1974 but is not equivalent in technical content. ISO 2501 is published by ISO. The main differences are that in the method described in ISO 2501 phenolphthalein is used as the indicator and the calculation of results includes a correction for the acidity of the sample.

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.

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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.

ii © BSI 12-1999

#### 1 Scope

This British Standard specifies requirements for butanone suitable for industrial purposes.

NOTE The titles of the publications referred to in this standard are listed on the inside back cover.

#### 2 Description

The material shall be clear and free from matter in suspension, as assessed by visual inspection, and shall consist essentially of butanone, CH<sub>3</sub>COC<sub>2</sub>H<sub>5</sub>.

### 3 Sampling and size of sample<sup>1)</sup>

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, or screw-capped bottle fitted with a polyethylene cone insert, of such capacity that it is almost filled by the sample.

NOTE 1 When it is necessary to seal the container, care should be taken to avoid the risk of contaminating the contents in any way.

NOTE 2 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.

#### 4 Colour

The colour of the material shall not exceed 10 Hazen units when measured by the method described in BS 5339.

#### 5 Density

The density of the material at 20  $^{\circ}$ C, determined by the method described in BS 4522, shall be not lower than 0.803 g/mL and not higher than 0.805 g/mL.

#### 6 Distillation range

When the material is distilled by the method described in BS 4591, modified as described in Appendix A, the initial boiling point at 1 013 mbar<sup>2)</sup> pressure shall be not lower than 79.0 °C and the dry point at 1 013 mbar pressure shall be not higher than 81.0 °C.

#### 7 Residue on evaporation

The residue on evaporation of the material shall not exceed 0.002 % (m/m) when determined by the method described in BS 4524.

#### 8 Water content

The material shall contain not more than 0.15% (m/m) of water when determined by one of the methods described in clause **5** of BS 2511:1970 using 20 mL of the material.

#### 9 Acidity

The acidity of the material, calculated as acetic acid (CH<sub>3</sub>COOH), shall not exceed 0.004 % (m/m) when determined by the method described in Appendix B.

#### 10 Alcoholic impurities

The material shall contain not more than 0.7 % (m/m) of alcoholic impurities, calculated as but anol ( $\rm C_4H_9OH$ ), when determined by the method described in Appendix C.

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<sup>&</sup>lt;sup>1)</sup> Detailed information on the sampling of liquid chemical products is given in BS 5309-1 and BS 5309-3.

 $<sup>^{2)}</sup>$  1 mbar = 100 N/m<sup>2</sup> = 100 Pa.

# Appendix A Determination of distillation range

Determine the distillation range, in terms of the initial boiling point and the dry point, by the method described in BS 4591, using the following thermometer, distillation conditions and temperature corrections.

- a) *Thermometer* (3.2 of BS 4591:1971). Use a thermometer designated F 100C/100 complying with BS 593.
- b) *Distillation* (**6.1** of BS 4591:1971). 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 (7.2 of BS 4591:1971). If the corrected barometric pressure deviates from 1 013 mbar apply corrections to the observed temperatures by subtracting 0.028 °C for every millibar above 1 013 mbar, or adding 0.028 °C for every millibar below 1 013 mbar.

NOTE These corrections are valid only for pressures above 933 mbar.

#### Appendix B 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 as indicator.

#### **B.2 Reagents**

- **B.2.1** *General*. During the analysis, use only reagents of recognized analytical grade, only methylated spirits complying with BS 3591, and only water complying with BS 3978.
- **B.2.2** *Sodium hydroxide*, standard volumetric solution, c(NaOH) = 0.100 mol/L.
- **B.2.3** *Phenolphthalein*, 5 g/L ethanolic solution. Dissolve 0.5 g of phenolphthalein in 100 mL of 95 % (V/V) ethanol, or 95 % (V/V) industrial methylated spirits, and add the sodium hydroxide solution (**B.2.2**) until a pale pink coloration is obtained.

NOTE 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 to ethanol.

#### **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, complying with class A of BS 846.

#### **B.4 Procedure**

#### **B.4.1** Test portion

Take  $100 \pm 1$  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. Cool slightly and then add the test portion (B.4.1). Boil the mixture gently for a further 5 min. At the end of this period, insert the stopper and allow to cool to ambient temperature. Remove the stopper, 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 coloration, persisting for about 15 s, is obtained. Stopper the flask and swirl its contents after each addition of sodium hydroxide solution.

#### **B.5** Expression of results

The acidity A, expressed as a percentage by mass of acetic acid (CH<sub>3</sub>COOH), is given by the equation:

$$A = \frac{0.006 \ V_1}{\rho}$$

where

- $V_1$  is the volume of the sodium hydroxide solution used for the determination (in mL);
- $\rho$  is the density of the sample at 20 °C (determined by the method described in BS 4522) (in g/mL);
- 0.006 is the mass of acetic acid corresponding to 1.00 mL of sodium hydroxide solution, c(NaOH) = 0.100 mol/L (in g).

# Appendix C Determination of alcoholic impurities

#### C.1 Principle

Any alcoholic impurities are acetylated by reaction with acetyl chloride. The amount of acetyl chloride that has reacted is determined by titration with standard volumetric sodium hydroxide solution, using 3,3-bis (4-hydroxy-1-naphthyl) phthalide (1-naphtholphthalein) as indicator, and the alcohol equivalent is calculated.

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#### C.2 Reagents

**C.2.1** *General*. During the analysis, use only reagents of recognized analytical grade, only methylated spirits complying with BS 3591, and only water complying with BS 3978.

C.2.2 Pyridine, dry.

**C.2.3** *Acetylating reagent.* To 118 mL of acetyl chloride, add sufficient dry toluene to give a total volume of 1 000 mL.

**C.2.4** *Sodium hydroxide*, standard volumetric solution, c(NaOH) = 1.00 mol/L.

C.2.5 3,3-bis(4-hydroxy-1-naphthyl)phthalide indicator solution, 5 g/L ethanolic solution.

Dissolve 0.5 g of 3,3-bis(4-hydroxy-1-naphthyl) phthalide in 100 mL of 95 % (*V/V*) ethanol or 95 % (*V/V*) industrial methylated spirits.

NOTE 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 to ethanol.

#### C.3 Apparatus

C.3.1 Ordinary laboratory apparatus

**C.3.2** *Two conical flasks*, of 250 mL capacity, fitted with ground glass stoppers.

C.3.3 *Pipette*, of 10 mL capacity, complying with class B of BS 1583, fitted with an automatic suction devices

**C.3.4** *Water bath*, capable of being maintained at  $60 \pm 1$  °C.

#### C.4 Procedure

#### C.4.1 Test portion

Take  $25 \pm 0.1$  mL of the sample, measured at 20 °C.

#### C.4.2 Determination

Dry the two conical flasks (C.3.2) and place 10.0 mL of the acetylating reagent (C.2.3) into each flask using the pipette (C.3.3). Add 2 mL of the pyridine (C.2.2) to each flask, immediately insert the stoppers tightly and mix the contents thoroughly without wetting the stoppers.

To one flask add the test portion (C.4.1), ensuring that all of it comes into contact with the acetylating reagent. Replace the stopper and mix the contents thoroughly without wetting the stopper.

Place both flasks in the water bath (C.3.4), maintained at  $60 \pm 1$  °C, loosening the stoppers momentarily to release any pressure and then replacing them tightly. Keep the flasks in the water bath for 20 min, shaking them occasionally, then remove the flasks and cool them to ambient temperature.

Add 25 mL of water and 0.5 mL of the indicator solution (C.2.5) to each flask and then titrate the contents of each flask in turn with the sodium hydroxide solution (C.2.4) until the appearance of a faint blue colour.

#### C.5 Expression of results

The alcohol content B, expressed as a percentage by mass of butanol ( $C_4H_9OH$ ), is given by the equation:

$$B = \frac{0.296 \; (V_1 - V_2)}{\rho}$$

where

 $V_1$  is the volume of the sodium hydroxide solution used for the titration of the blank (in mL);

 $V_2$  is the volume of the sodium hydroxide solution used for the titration of the test solution (in mL);

ho is the density of the sample at 20 °C (determined by the method described in BS 4522) (in g/mL).

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4 blank

## Publications referred to

BS 593, Laboratory thermometers.

BS 846, Specification for burettes.

BS 1583, One-mark pipettes.

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

BS 3591, Specification for industrial methylated spirits.

BS 3978, Water for laboratory use.

BS 4522, Method for the determination of density of liquids at 20 °C.

 $BS\ 4524$ ,  $Method\ for\ the\ determination\ of\ residue\ on\ evaporation\ on\ a\ water\ bath.$ 

BS 4591, Method for the determination of distillation characteristics.

BS 5309, Methods for sampling chemical products.

BS 5309-1, Introduction and general principles.

BS 5309-3, Sampling of liquids.

BS 5339, Method of measurement of colour in Hazen units (platinum-cobalt scale) of liquid chemical products.

ISO 2497, Methyl ethyl ketone for industrial use — List of methods of test<sup>3)</sup>.

ISO 2501, Methyl ethyl ketone, isobutyl methyl ketone, and isoamyl ethyl ketone for industrial use — Determination of alcoholic impurities — Volumetric method<sup>3)</sup>.

ISO 2887, sec Butyl alcohol, methyl ethyl ketone, isobutyl methyl ketone, isoamyl ethyl ketone, diacetone alcohol, and hexylene glycol for industrial use — Determination of acidity to phenolphthalein — Volumetric method<sup>3)</sup>.

<sup>&</sup>lt;sup>3)</sup> Referred to in the foreword only.

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