Specification for

4-methylpentan-2-one (isobutyl methyl ketone) for industrial use

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Committees responsible for this British Standard

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British Pharmacopoeia Commission
British Society of Perfumers
Chemical Industries' Association
Oil and Colour Chemists' Association
Royal Society of Chemistry
Solvents Industry Association Ltd.

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Content

Foreword

This British Standard, which has been prepared under the direction of the Chemicals Standards Policy Committee, comprises a specification for 4-methylpentan-2-one to meet the requirements of a wide range of industrial users.

This British Standard supersedes BS 1941:1968 which is withdrawn. In this revision of BS 1941 the requirements for water content and alcoholic impurities have been made more stringent and the requirement for relative density has been replaced by one for density at 20 °C.

This British Standard is related to ISO 2499:1974 but is not equivalent in technical content. ISO 2499 is published by the International Organization for Standardization (ISO) and lists various general test methods and test methods specifically for 4-methylpentan-2-one. Several of the methods listed in ISO 2499 correspond to those which are referred to in this British Standard. ISO 2499 however does not specify limits for any of the properties of 4-methylpentan-2-one.

Annex B of this British Standard is related to ISO 2887:1973, published by ISO, but is not equivalent in technical content. 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.

Annex C of this British Standard is related to ISO 2501:1974, published by ISO, but is not equivalent in technical content. 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.

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, 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.

1 Scope

This British Standard specifies requirements for 4-methylpentan-2-one (isobutyl methyl ketone) suitable for industrial purposes.

2 References

2.1 Normative references

This British Standard incorporates, by reference, provisions from specific editions of other publications. These normative references are cited at the appropriate points in the text and the publications are listed on the inside back cover. Subsequent amendments to, or revisions of, any of these publications apply to this British Standard only when incorporated in it by updating or revision.

2.2 Informative references

This British Standard refers to other publications that provide information or guidance. Editions of these publications current at the time of issue of this standard are listed on the inside back cover, but reference should be made to the latest editions.

3 Description

The material shall be clear and free from matter in suspension, as assessed by visual inspection, and shall consist essentially of 4-methylpentan-2-one, $CH_3COCH_2CH(CH_3)_2$.

4 Sampling and size of sample[1)]

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 airtight, 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.

5 Colour

The colour of the material shall not exceed 10 Hazen units when measured by the method described in BS 5339:1976.

6 Density

The density of the material at 20 °C, determined by the method described in BS 4522:1988, shall be not lower than 0.799 g/ml and not higher than 0.802 g/ml.

7 Distillation range

When the material is distilled by the method described in BS 4591:1990, modified as described in Annex A of this standard, the initial boiling point at 101.3 kPa [2)] pressure shall be not lower than 114.0 °C and the dry point at 101.3 kPa pressure shall be not higher than 117.0 °C.

8 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 4524:1983.

9 Water content

The material shall contain not more than 0.10 % (*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.

10 Acidity

The acidity of the material, calculated as acetic acid (CH_3COOH), shall not exceed 0.0020 % (*m*/*m*) when determined by the method described in Annex B.

11 Alcoholic impurities

The material shall contain not more than 0.30 % (*m*/m) of alcoholic impurities, calculated as hexanol ($C_6H_{13}OH$), when determined by the method described in Annex C.

[1)] Detailed information on the sampling of liquid chemical products is given in BS 5309-1:1976 and BS 5309-3:1976.
[2)] 1 kPa = 10 mbar.

Annex A (normative) 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 4591:1990, using the following thermometer, distillation conditions and temperature corrections.

a) *Thermometer* (see **5.1.2** of BS 4591:1990). Use a thermometer designated F150C/100 conforming to BS 593:1989.

b) *Distillation* (see **7.2** of BS 4591:1990). 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* (see **9.1.2** of BS 4591:1990). If the corrected barometric pressure deviates from 1 013 mbar apply corrections to the observed temperatures by subtracting 0.034 °C for every millibar above 1 013 mbar, or adding 0.034 °C for every millibar below 1 013 mbar.

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

Annex B (normative) 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 conforming to BS 3591:1985, and only water conforming to grade 3 of BS 3978:1987.

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 The use of industrial methylated spirits is governed by The Methylated Spirits Regulations, 1983 (S.I. 1983 No. 252) [1]. It is not permissible to use duty-free ethanol, received under the provisions of The Alcoholic Liquor Duties Act 1979, Section 10 [2], 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, conforming to class A of BS 846:1985.

B.4 Procedure

B.4.1 ***Test portion***

Take 100 ml ± 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_3COOH), 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);

ρ is the density of the sample at 20 °C (determined by the method described in BS 4522:1988) (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).

Report the value of A in % (m/m) to two significant figures.

Annex C (normative) 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.

C.2 Reagents

C.2.1 *General*

During the analysis, use only reagents of recognized analytical grade, only methylated spirits conforming to BS 3591:1985, and only water conforming to grade 3 of BS 3978:1987.

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 The use of industrial methylated spirits is governed by The Methylated Spirits Regulations, 1983 (S.I. 1983 No. 252) [1]. It is not permissible to use duty-free ethanol, received under the provisions of The Alcoholic Liquor Duties Act 1979, Section 10 [2], 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, conforming to class B of BS 1583:1986, fitted with an automatic suction device.

C.3.4 *Water bath*, capable of being maintained at 60 °C ± 1 °C.

C.4 Procedure

C.4.1 ***Test portion***

Take 25 ml ± 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 °C ± 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 hexanol ($C_6H_{13}OH$), is given by the equation:

$$B = \frac{0.408\ (V_1 - V_2)}{\rho}$$

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);

ρ is the density of the sample at 20 °C (determined by the method described in BS 4522:1988) (in g/ml).

Report the value of B in % (m/m) to two significant figures.

blank

List of references (see clause 2)

Normative references

BSI standards publications

BRITISH STANDARDS INSTITUTION, London

BS 593:1989, *Specification for laboratory thermometers.*

BS 846:1985, *Specification for burettes.*

BS 1583:1986, *Specification for one-mark pipettes.*

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

BS 3591:1985, *Specification for industrial methylated spirits.*

BS 3978:1987, *Specification for water for laboratory use.*

BS 4522:1988, *Method for determination of absolute density at 20 °C of liquid chemical products for industrial use.*

BS 4524:1983, *Method for determination of residue on evaporation on a water bath.*

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

BS 5339:1976, *Method of measurement of colour in Hazen units (platinum-cobalt scale) of liquid chemical products.*

Informative references

BSI standards publications

BRITISH STANDARDS INSTITUTION, London

BS 5309, *Methods for sampling chemical products.*

BS 5309-1:1976, *Introduction and general principles.*

BS 5309-3:1976, *Sampling of liquids.*

ISO standards publications

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION (ISO), Geneva. (All publications are available from BSI Sales).

ISO 2499:1974, *isoButyl methyl ketone for industrial use — List of methods of test*[3)].

ISO 2501:1974, *Methyl ethyl ketone, isobutyl methyl ketone and isoamyl ethyl ketone for industrial use — Determination of alcoholic impurities — Volumetric method*[3)].

ISO 2887:1973, *secButyl 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*[3)].

Other references

[1] GREAT BRITAIN. The Methylated Spirits Regulations, 1983. London: HMSO.

[2] GREAT BRITAIN. The Alcoholic Liquor Duties Act, 1979. London: HMSO.

[3)] Referred to in the foreword only.

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