

# Propan-2-ol (isopropyl alcohol) for industrial use —

## Part 2: Methods of test

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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/4 upon which the following bodies were represented:

Chemical Industries' Association  
 Ministry of Defence  
 Oil and Colour Chemists' Association  
 Royal Society of Chemistry  
 Society of Chemical Industry

The following bodies were also represented in the drafting of the standard, through subcommittees and panels:

British Pharmacopoeia Commission  
 British Society of Perfumers  
 Cosmetic, Toiletry and Perfumery Association Limited  
 Department of Trade and Industry (Laboratory of the Government Chemist)

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## Foreword

This Part of this British Standard, which has been prepared under the direction of the Chemicals Standards Committee, provides a comprehensive series of test methods for propan-2-ol for industrial use. Although applicable to the material in general, it includes all the test methods required to assess compliance with BS 1595-1. The standard previously numbered BS 1595 (published in 1965) has been amended to become BS 1595-1 “*Specification*” and the methods of test that were included in the appendices have been deleted.

In preparing this standard, the opportunity has been taken to implement Parts 1, 2 and 3 of ISO 756 “*Propan-2-ol for industrial use — Methods of test*”, published by the International Organization for Standardization (ISO). These standards have been prepared, with the active participation of the UK, by Technical Committee 47, Chemistry, of the International Organization for Standardization (ISO), as separate parts of ISO 756 and constitute the revision of ISO Recommendation R 756. Subsequent International Standards in the ISO 756 series, if approved by the UK, will be published as additions to this British Standard, without technical alteration.

Clauses 2 to 8 and clause 11 of this British Standard are related to ISO 756-1 but not equivalent in technical content. The technical differences occur in the method for the determination of aldehydes and ketones content (see clause 8): in this British Standard only one concentration of potassium hydroxide solution is allowed for the titration and only one form of expression of the results (as a percentage by mass calculated as acetone) is given. ISO 756-1 refers to the general method described in ISO 1843-3 for the determination of aldehydes and ketones content, and gives the additional information needed to carry out the determination for propan-2-ol: in this British Standard, the method is described in full.

Clause 9 of this standard, which describes a method for the determination of acidity, is equivalent to ISO 756-2 in technical content but differs in presentation.

Clause 10 of this standard, which describes a test for miscibility with water, is equivalent to ISO 756-3 in technical content but differs in presentation.

**This Part of this standard describes methods of test only and should not be referred to as a specification defining limits of purity. Reference to this Part of this standard should indicate that the methods of test used are in accordance with the appropriate clause(s) of BS 1595-2.**

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 Part of this British Standard describes methods for testing propan-2-ol for industrial use.

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

## 2 Sampling<sup>1)</sup>

Store the laboratory sample in a clean, dry and airtight, ground glass stoppered bottle, or a screw-capped bottle fitted with a polyethylene cone insert, of such capacity that it is almost filled by the sample. 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. If it is necessary to seal the bottle, take care to avoid any risk of contamination of the contents. Store the sample in a cool place in the dark.

## 3 Determination of density at 20 °C

Determine the density at 20 °C by the method described in BS 4522.

## 4 Determination of distillation characteristics

Determine the distillation characteristics by the method described in BS 4591, except that the following thermometer, temperature corrections and distillation conditions shall be used.

- a) *Thermometer* (3.2 of BS 4591:1971). Use a thermometer designated F 100C/100 complying with the requirements of BS 593.
- b) *Corrections to be applied to observed temperatures* (7.2 of BS 4591:1971). If the corrected barometric pressure deviates from 1 013 mbar<sup>2)</sup> apply a correction to the observed temperature by subtracting 0.025 °C for every millibar above, or adding 0.025 °C for every millibar below, 1 013 mbar.
- c) *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 7 to 12 min.

## 5 Determination of residue on evaporation on a water bath

Determine the residue on evaporation by the method described in BS 4524.

## 6 Measurement of colour

Determine the colour by the method described in BS 5339.

## 7 Determination of water content

Determine the water content by one of the methods described in BS 2511.

## 8 Determination of aldehydes and ketones content

NOTE This method is based on ISO 1843-3 which is to be implemented as part of the revision of BS 4583:1970.

### 8.1 Principle

Reaction of carbonyl compounds present in a test portion with hydroxylammonium chloride to form an oxime, and potentiometric titration of the hydrochloric acid liberated with standard volumetric ethanolic potassium hydroxide solution.

### 8.2 Reagents

**8.2.1 General.** During the analysis, use only reagents of recognized analytical grade and only water complying with BS 3978.

NOTE The ethanol specified in 8.2.2 and 8.2.4 may be replaced for these purposes by industrial methylated spirits, 95 % (V/V) complying with 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 to ethanol.

**8.2.2 Hydroxylammonium chloride**, 10 g/L ethanolic solution.

Dissolve 50 g of hydroxylammonium chloride (NH<sub>2</sub>OH.HCl) in 90 mL of water and dilute to 1 000 mL with 95 % (V/V) ethanol. Further dilute 100 mL of this solution to 500 mL with 95 % (V/V) ethanol.

**WARNING.** Hydroxylammonium chloride is toxic, corrosive and an irritant. Avoid contact with eyes and skin.

<sup>1)</sup> Additional guidance is given in BS 5309-1 and BS 5309-3.

<sup>2)</sup> 1 mbar = 100 N/m<sup>2</sup> = 100 Pa.

**8.2.3 Ethanol**, anhydrous, carbonyl-free, prepared as follows.

Boil under reflux 500 mL of anhydrous ethanol with 5 g of 2,4-dinitrophenylhydrazine and 5 drops of hydrochloric acid solution,  $\rho$  approximately 1.19 g/mL, for 2 to 3 h. Distil off the ethanol slowly using a Widmer distillation column about 300 mm long and about 25 mm in diameter, or any other suitable column. Reject the first 50 mL of distillate and collect the next 400 mL, rejecting the remainder. If the distillate is found to be coloured, carry out a redistillation.

**8.2.4 Potassium hydroxide**, standard volumetric solution in 95 % (V/V) ethanol,  $c$  (KOH) = 0.1 mol/L.

### 8.3 Apparatus

**8.3.1 Ordinary laboratory apparatus**

**8.3.2 Two conical flasks**, of borosilicate glass, of capacity 250 mL, fitted with ground glass stoppers.

**8.3.3 Two water-cooled reflux condensers**, with ground glass joints to fit the flasks (8.3.2).

**8.3.4 pH meter**, fitted with a glass measuring electrode and a calomel reference electrode.

### 8.4 Procedure

**8.4.1 Test portion.** Into one of the conical flasks (8.3.2) already containing 10 mL of the hydroxylammonium chloride solution (8.2.2) weigh, to the nearest 0.001 g, 25 g to 30 g of the laboratory sample.

**8.4.2 Blank test.** Carry out a blank test at the same time as the determination, following the same procedure but omitting the test portion.

**8.4.3 Determination.** Add 10 mL of the ethanol (8.2.3) to the conical flask (8.3.2) containing the test portion (8.4.1). Attach one of the condensers (8.3.3) to the flask and reflux for 30 min on a boiling water bath. Remove the flask, still carrying its condenser, from the boiling water bath and allow to cool to ambient temperature. When cold, wash down the inside of the condenser with 10 mL of the ethanol. Transfer the contents of the flask quantitatively to a 400 mL beaker, washing with 125 mL of the ethanol.

Titrate with the potassium hydroxide solution (8.2.4) using the pH meter (8.3.4). The volume/potential graph may be plotted directly, in which case the point of inflection corresponds to the end-point of the titration (pH value about 3). Alternatively, the first derived curve may be plotted, in which case the end-point of the titration corresponds to the turning point of the curve.

### 8.5 Expression of results

The aldehydes and ketones content, expressed as a percentage by mass of acetone ( $\text{CH}_3\text{COCH}_3$ ), is given by the formula

$$\frac{0.58 (V_1 - V_0)}{m}$$

where

$V_0$  is the volume of the potassium hydroxide solution used for the blank test (in mL);

$V_1$  is the volume of the potassium hydroxide solution used for the determination (in mL);

$m$  is the mass of the test portion (in g).

## 9 Determination of acidity

### 9.1 Field of application

This method is applicable to products having an acidity, expressed as acetic acid ( $\text{CH}_3\text{COOH}$ ), equal to or greater than 0.0006 % (m/m).

### 9.2 Principle

Dilution of a test portion with carbon dioxide-free water. Titration of the acidity with standard volumetric sodium hydroxide solution, in the presence of phenolphthalein as indicator.

### 9.3 Reagents

**9.3.1 General.** During the analysis, use only reagents of recognized analytical grade and only carbon dioxide-free water recently prepared in accordance with 9.3.2.

**9.3.2 Water, carbon dioxide-free.** Boil water, complying with BS 3978, and allow it to cool in a flask fitted with a stopper carrying a soda-lime guard-tube.

**9.3.3 Sodium hydroxide**, standard volumetric solution,  $c$ (NaOH) = 0.1 mol/L.

**9.3.4 Phenolphthalein**, 5 g/L ethanolic solution.

Dissolve 0.5 g of phenolphthalein in 100 mL of 95 % (V/V) ethanol, and add the sodium hydroxide solution (9.3.3) until a pale pink colouration is obtained.

**NOTE** The ethanol may be replaced for this purpose by industrial methylated spirits, 95 % (V/V) complying with 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 to ethanol.

## 9.4 Apparatus

**9.4.1 Ordinary laboratory apparatus**

**9.4.2 Conical flask**, of borosilicate glass, of capacity 500 mL, fitted with a ground glass stopper carrying a soda-lime guard-tube.

**9.4.3 Burette**, of capacity 10 mL, graduated in 0.02 mL divisions, complying with class A of BS 846.

## 9.5 Procedure

**9.5.1 Test portion.** Take  $100 \pm 0.1$  mL of the laboratory sample at a temperature of about 20 °C.

**9.5.2 Determination.** Place 100 mL of the water (9.3.2) in the conical flask (9.4.2), add 0.5 mL of the phenolphthalein solution (9.3.4) and restore the pale pink colouration by the addition of one drop of the sodium hydroxide solution (9.3.3). Add the test portion (9.5.1), and a further 0.5 mL of the phenolphthalein solution. Titrate the test solution with the sodium hydroxide solution (9.3.3), stoppering the flask and swirling its contents after each addition, until a pale pink coloration, persisting for about 15 s, is obtained.

## 9.6 Expression of results

The acidity, expressed as a percentage by mass of acetic acid ( $\text{CH}_3\text{COOH}$ ), is given by the formula

$$\frac{0.006 V}{\rho}$$

where

$V$  is the volume of the sodium hydroxide solution used for the determination (in mL);

$\rho$  is the density of the sample at 20 °C (in g/mL) (see clause 3);

0.006 is the mass (in g) of acetic acid corresponding to 1 mL of sodium hydroxide solution,  
 $c$  (NaOH) = 0.100 mol/L.

**NOTE** If the standard volumetric solution used does not have the exact concentration stated in the list of reagents, an appropriate correction should be applied.

Report the result to two significant figures.

## 10 Test for miscibility with water

### 10.1 Principle

Addition of water to a test portion, under specified conditions, and examination for any opalescence or turbidity.

## 10.2 Reagent

During the test, use only water complying with BS 3978.

## 10.3 Apparatus

**10.3.1 Ordinary laboratory apparatus**

**10.3.2 Two matched Nessler cylinders**, of capacity 100 mL, similar in every respect, complying with BS 612.

## 10.4 Procedure

**10.4.1 Test portion.** Take, by means of a safety pipette, 5 mL of the laboratory sample at a temperature of about 20 °C and introduce it into one of the Nessler cylinders (10.3.2).

**10.4.2 Test.** Add slowly, with thorough mixing, 95 mL of water to the test portion (10.4.1) in the Nessler cylinder. Examine for opalescence during the addition. Adjust the temperature of the mixture to 20 °C.

Examine vertically for opalescence or turbidity against a black background with side illumination, using as a standard the second Nessler cylinder containing 100 mL of water.

## 10.5 Expression of results

Report the occurrence of opalescence or turbidity or whether the solution remained clear.

## 11 Test reports

The test report, for each determination, should contain the following:

- an identification of the sample;
- a reference to the method used;
- the results, and the method of expression used;
- any unusual features noted during the determination;
- any operation not included in the appropriate clause of this standard or in the British Standards to which reference is made, or regarded as optional.







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## Publications referred to

- BS 593, *Laboratory thermometers.*
- BS 612, *Nessler cylinders.*
- BS 846, *Specification for burettes.*
- BS 2511, *Methods for the determination of water (Karl Fischer method).*
- BS 3591, *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 determination of residue on evaporation on a water bath.*
- BS 4583, *Methods of test for higher alcohols.*
- 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 756, *Propan-2-ol for industrial use — Methods of test.*
- ISO 756-1, *General*<sup>3)</sup>.
- ISO 756-2, *Determination of acidity — Titrimetric method*<sup>1)</sup>.
- ISO 756-3, *Test for miscibility with water*<sup>1)</sup>.
- ISO 1843-3, *Higher alcohols for industrial use — Methods of test — Part III: Determination of carbonyl compounds content — Potentiometric method.*

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

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