

Methods of test for

Higher alcohols 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 Policy Committee (CIC/-) to Technical Committee CIC/51, upon which the following bodies were represented:

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

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Foreword

This British Standard has been prepared under the direction of the Chemicals Standards Policy Committee and provides a comprehensive series of test methods for C₆ to C₁₃ alcohols for industrial use.

This revision supersedes BS 4583:1970, which is withdrawn.

The principal differences between this standard and the 1970 edition are as follows.

- a) The scope has been extended to include C₆ alcohols.
- b) General test methods are now specified for the measurement of colour (see BS 5339) and the determination of distillation characteristics (see BS 4591).
- c) For the determination of ash (see clause 7), the heating in a furnace is carried out at a temperature of 600 ± 30 °C instead of 630 ± 30 °C.
- d) Acidity is now expressed as a percentage by mass of acetic acid instead of milliequivalents per kilogram.
- e) Aldehydes and ketones are now determined by titrating the liberated hydrochloric acid, from the reaction with hydroxylammonium chloride, with ethanolic potassium hydroxide solution (see clause 9).
- f) For the measurement of colour after treatment with sulphuric acid (see clause 10), the reaction vessel is now cooled in an ice-water bath before, during and immediately after the addition of the sulphuric acid solution.
- g) For the determination of total alcohols content (see clause 11), acetic anhydride is used for the esterification, instead of phthalic anhydride, and the results are now expressed as a percentage by mass.
- h) The determination of bromine number (see clause 12) has been added.

In preparing this standard, account has been taken of Parts 1 to 8 of ISO 1843 “*Higher alcohols for industrial use — Methods of test*”, published by the International Organization for Standardization (ISO). Appendix A gives the relationship between international standards and this British Standard, together with the relationship between general test methods and corresponding international standards.

This standard describes methods of test only and should not be used or quoted as a specification defining limits of purity. Reference to this standard should indicate that the methods of test used are in accordance with the appropriate clauses of BS 4583:1991.

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 8, 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 describes methods for testing C₆ to C₁₃ alcohols for industrial use.

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

2 Sampling¹⁾

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 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 Measurement of colour

Determine the colour by the method described in BS 5339.

4 Determination of density at 20 °C

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

5 Determination of distillation characteristics

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

- a) *Thermometer* (see 5.1.2 of BS 4591:1990). For measurement of distillation ranges not greater than 5 °C, use a partial immersion thermometer, graduated at intervals of 0.2 °C, complying with BS 593 (e.g. a thermometer designated F200C/100). For measurement of distillation ranges greater than 5 °C, use a thermometer designated F255C/100 complying with BS 593.
- 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 15 min to 20 min.
- c) *Corrections to be applied to observed temperatures* (see 9.1.2 of BS 4591:1990). If the corrected barometric pressure deviates from 101.3 kPa apply corrections to the observed temperatures by subtracting 0.038 °C for every 0.1 kPa above 101.3 kPa, or adding 0.038 °C for every 0.1 kPa below 101.3 kPa.

6 Determination of water content

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

7 Determination of ash

7.1 Principle

A test portion is burned and then heated at a temperature of 600 ± 30 °C to constant mass.

7.2 Apparatus

7.2.1 *Ordinary laboratory apparatus.*

7.2.2 *Platinum or silica dish.*

7.2.3 *Electric furnace*, capable of being controlled at a temperature of 600 ± 30 °C.

7.3 Procedure

7.3.1 Test portion

Weigh, to the nearest 0.1 g, about 50 g of the laboratory sample.

7.3.2 Determination

In the dish (see 7.2.2), previously heated at a temperature of 600 ± 30 °C, cooled in a desiccator and weighed to the nearest 0.0001 g, slowly burn the test portion (see 7.3.1) in several portions. Heat finally in the furnace (see 7.2.3), controlled at 600 ± 30 °C, until all the carbonaceous matter has disappeared. Allow to cool in a desiccator and weigh to the nearest 0.0001 g. Repeat the operations of heating, cooling and weighing until the difference in mass between two successive weighings does not exceed 0.0005 g.

7.4 Expression of results

The ash *A*, expressed as a percentage by mass, is given by the equation:

$$A = \frac{100 \ m_1}{m_0}$$

where

*m*₀ is the mass of the test portion (see 7.3.1) (in g);

*m*₁ is the mass of the residue (in g).

8 Determination of acidity

8.1 Principle

The acidity in a test portion is titrated with a standard volumetric sodium hydroxide solution, in ethanolic medium, using phenolphthalein as indicator.

¹⁾ Detailed information on the sampling of liquid chemical products is given in BS 5309-1 and BS 5309-3.