BRITISH STANDARD

BS 6392-6: 1983

Incorporating Amendment No.1

Testing of ethanol for industrial use —

Part 6: Method for determination of methanol content [0.01 % (*V*/*V*) to 0.20 % (*V*/*V*)] (photometric method)

NOTE It is recommended that this Part be read in conjunction with the information given in the "*General introduction*" published separately as BS 6392-0.

Confirmed January 2011

UDC 661.722:543.42.062:547.261

Foreword

This Part of BS 6392 is technically equivalent to ISO 1388 "*Ethanol for industrial use — Methods of test*" Part 7 "*Determination of methanol content [methanol contents between 0,01 and 0,20 % (V/V)] — Photometric method*", published in 1981 by the International Organization for Standardization (ISO).

For ease of production, the text of ISO 1388-7:1981, with the omission of the Annex, has been used for this British Standard. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

In **3.3.2** reference is made to a sintered glass filter funnel grade number 4 A complying with BS 1752. When BS 1752 is revised it is expected that it will be identical with ISO 4793 in which the equivalent to grade 4 A of BS 1752:1963 is porosity grade P 10.

This standard describes a method only and should not be used as a specification defining limits of purity. Reference to the standard should indicate that the method of test used is in accordance with BS 6392-6.

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 and 2, 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.

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 31 August 1983

The Committees responsible for this British Standard are shown in Part 0.
The following BSI references relate to the work on this standard:
Committee reference CIC/4
Draft for comment 80/51209 DC

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Amendments issued since publication

Amd. No.	Date of issue	Comments
4942	March 1986	Indicated by a sideline in the margin

Contents

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1 Scope and field of application

This Part of BS 6392 describes a photometric method for the determination of the methanol content of ethanol for industrial use.

The method is applicable to products having methanol contents between 0,01 and 0,20 % (*V*/*V*).

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

2 Principle

Conversion of the methanol present in a test portion to formaldehyde by oxidation with a solution of potassium permanganate in phosphoric acid. Reaction of the formaldehyde formed with chromotropic acid.

Photometric measurement of the violet coloration obtained at a wavelength of about 570 nm.

3 Reagents

During the analysis, use only reagents of recognized analytical grade, and distilled water or water of equivalent purity.

3.1 *Potassium permanganate*, 30 g/l solution in phosphoric acid.

Dissolve 3 g of potassium permanganate in a little water, add 15,5 ml of orthophosphoric acid solution, ϱ 1,69 g/ml, dilute to 100 ml with water and mix.

3.2 *Disodium disulphite* [sodium metabisulphite ($Na_2S_2O_5$)], 100 g/l solution.

Dissolve 10 g of sodium metabisulphite in water and dilute to 100 ml.

3.3 *4,5-Dihydroxynaphthalene-2,7-disulphonic acid* (chromotropic acid), solution in sulphuric acid.

3.3.1 *Preparation of the solution*

Dissolve 0,1 g of chromotropic acid, or its *di*sodium salt, in 10 ml of water. Add, while cooling, 90 ml of sulphuric acid, ϱ approximately 1,81 g/ml, about 90 % (*m*/*m*) solution, and mix.

Prepare this solution at the time of use.

If the solution causes significant coloration during colour development of the compensation solution (**5.3.1**) or the blank test solution (**5.2**), purify the chromotropic acid, or its *di*sodium salt, in accordance with the procedure specified in **3.3.2**.

3.3.2 *Purification of the chromotropic acid*

Dissolve about 10 g of chromotropic acid, or its *di*sodium salt, in 25 ml of water. If the *di*sodium salt is used, add, while cooling, 2 ml of sulphuric acid, ϱ approximately 1,84 g/ml, to convert it to the free acid. Add 50 ml of methanol, heat just to boiling and filter through a sintered glass filter funnel, grade number 4 A complying with BS 1752.

Add 100 ml of propan-2-ol to the solution to precipitate the chromotropic acid. Collect the precipitate on a sintered glass filter funnel, grade number 4 A complying with BS 1752 and wash it with small portions of propan-2-ol. Allow to dry, initially in air, and finally in a desiccator over sulphuric acid, ϱ approximately 1,84 g/ml, about 98 % (*m*/*m*) solution, as desiccant.

If, after purification, the blank test solution is still coloured, reject the chromotropic acid.

3.4 *Methanol*, standard solution corresponding to 0,05 % (*V*/*V*) of methanol.

Place 1,00 ml of absolute methanol in a 250 ml one-mark volumetric flask, add a quantity of methanol-free ethanol corresponding to 99 ml of anhydrous ethanol, dilute to the mark with water and mix.

Place 25,0 ml of this solution in a 200 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 0,000 5 ml of methanol.

NOTE Industrial methylated spirits 95 % (*V*/*V*) is not suitable for use in place of the methanol-free ethanol used in the preparation of this reagent.

4 Apparatus

Ordinary laboratory apparatus, and

4.1 *Water bath*, capable of being controlled at 70 ± 2 °C.

4.2 *Spectrophotometer*, or

4.3 *Photoelectric absorptiometer*, fitted with filters ensuring maximum transmission in the region of 570 nm.

5 Procedure

NOTE Industrial methylated spirits 95 % (*V*/*V*) is not suitable for use in place of the ethanol specified in **5.2**, **5.3.1**, **5.3.4** and **5.4.2**.

5.1 Test portion and preparation of the test solution

Take as the test portion a volume (V_2) of the laboratory sample corresponding to 5,0 ml of anhydrous ethanol and place it in a 100 ml one-mark volumetric flask. Prepare the test solution by diluting to the mark with water and mixing.

5.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all the reagents used for the determination, but replacing the test portion by a volume of methanol-free ethanol corresponding to 5,0 ml of anhydrous ethanol.

5.3 Preparation of the calibration graph

5.3.1 Preparation of standard solutions, used for the preparation of standard colorimetric solutions

Into a series of six 100 ml one-mark volumetric flasks, place the volumes of the standard methanol solution (**3.4**) indicated in the following table, dilute to the mark with a 5 % (*V*/*V*) solution of methanol-free ethanol in water, and mix.

Standard methanol solution (3.4)	Corresponding volume of methanol
ml	ml
0[a]	0
1,00	0,000 5
2,50	0,001 25
5,00	0,002 5
10,00	0,005
20,00	0,010

[a] Compensation solution.

5.3.2 Preparation of standard colorimetric solutions, for photometric measurements carried out in cells of optical path length 1 cm

Into a series of six test tubes, place 2,0 ml of each of the dilute standard methanol solutions (**5.3.1**).

5.3.3 ***Colour development***

Add, to each tube, 1,0 ml of the potassium permanganate solution (**3.1**), and, after 15 min, 0,6 ml of the *di*sodium disulphite solution (**3.2**). To these solutions, which shall be colourless, add, while cooling with ice, 10,0 ml of the chromotropic acid solution (**3.3**), and heat on the water bath (**4.1**), controlled at 70 ± 2 °C, for 20 min. Remove the tubes from the water bath and allow to cool.

NOTE 1 If the colour of the compensation solution is more intense than that of the most dilute standard colorimetric solution, purify the chromotropic acid, or its *di*sodium salt, as specified in **3.3.2**.

NOTE 2 Prepare a new calibration graph each time a new bottle of chromotropic acid is used.

5.3.4 ***Photometric measurements***

Carry out photometric measurements on each standard colorimetric solution (**5.3.2**) using either the spectrophotometer (**4.2**), set at a wavelength of about 570 nm, or the photoelectric absorptiometer (**4.3**) fitted with appropriate filters, after having adjusted the instrument to zero absorbance against a 5 % (*V*/*V*) solution of ethanol in water.

5.3.5 ***Plotting the graph***

Deduct the absorbance of the compensation solution from those of the standard colorimetric solutions (**5.3.2**). Plot a graph having, for example, the volumes, in millilitres, of methanol in the standard solutions (**5.3.1**) as abscissae and the corresponding corrected values of absorbance as ordinates.

5.4 Determination

5.4.1 ***Colour development***

Take 2,0 ml of the test solution (**5.1**), place it in a test tube and proceed as specified in **5.3.3**.

5.4.2 ***Photometric measurements***

Carry out photometric measurements on the test solution and on the blank test solution, proceeding as specified in **5.3.4**, after having adjusted the instrument to zero absorbance against a 5 % (*V*/*V*) solution of ethanol in water.

6 Expression of results

By means of the calibration graph (**5.3.5**), determine the volumes of methanol corresponding to the values of the photometric measurements.

The methanol content, expressed as methanol as a percentage by volume, is given by the formula

$$\frac{(V_1 - V_0) \times 100}{V_2}$$

where

V_0 is the volume, in millilitres, of methanol found in the blank test solution;

V_1 is the volume, in millilitres, of methanol found in the test solution;

V_2 is the volume, in millilitres, of the test portion.

Publications referred to

BS 1752, *Laboratory sintered or fritted filters.*
ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*[1)].

[1)] Referred to in the foreword only.

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W4 4AL