BRITISH STANDARD

BS 6392-7: 1983

Incorporating Amendment No. 1

Testing of ethanol for industrial use —

Part 7: Method for determination of methanol content [0.10 % (*V*/*V*) to 1.50 % (*V*/*V*)] (visual colorimetric 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 661.722:543.432.062:547.261

Foreword

This Part of BS 6392 is technically equivalent to ISO 1388, "*Ethanol for industrial use — Methods of test*" Part 8 "*Determination of methanol content [methanol contents between 0,10 and 1,50 % (V/V)] — Visual colorimetric method*", published in 1981 by the International Organization for Standardization (ISO).

For ease of production, the text of ISO 1388-8:1981, 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.

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 6392-7.

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 and 2 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
4943	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 visual colorimetric method for the determination of the methanol content of ethanol for industrial use.

The method is applicable to products having methanol contents between 0,10 and 1,50 % (*V*/*V*).

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 Schiff reagent. Visual comparison of the colour obtained with the colours of standard colorimetric solutions containing known quantities of formaldehyde.

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 *Oxalic acid*, 50 g/l solution in sulphuric acid.

WARNING — Harmful in contact with skin and if swallowed. Avoid contact with skin and eyes.

Dissolve 5 g of oxalic acid in 100 ml of 50 % (*V*/*V*) sulphuric acid solution, prepared by diluting sulphuric acid, ϱ approximately 1,84 g/ml, about 98 % (*m*/*m*) solution, 1 + 1 (*V*/*V*) with water.

3.3 *Schiff reagent*

WARNING — Basic fuchsin is carcinogenic. Avoid skin contact with basic fuchsin and its solutions and inhalation of its dust.

3.3.1 *Preparation*

Place 1 500 ml of water in a 3 000 ml conical flask, add 4,500 ± 0,005 g of *p*-rosaniline hydrochloride (basic fuchsin) and swirl to dissolve.
Add 9,6 ± 0,05 g of *di*sodium disulphite [sodium metabisulphite ($Na_2S_2O_5$)], mix, and allow to stand for 5 to 10 min. Add 40 ml of approximately 295 g/l sulphuric acid solution, mix thoroughly, stopper the flask and allow to stand for about 12 h. Decolorize the solution, if necessary, by treatment with activated carbon.

3.3.2 ***Determination and adjustment of free sulphur dioxide content***

Transfer 10 ml of the colourless reagent (**3.3.1**) to a 250 ml conical flask. Add 20 ml of water and 5 ml of freshly prepared starch solution and titrate the solution with standard volumetric iodine solution, $c(1/2\ I_2)$ = 0,1 mol/l, until the characteristic dark blue colour is just obtained.

NOTE 1 ml of iodine solution, $c(1/2\ I_2)$ = 0,1 mol/l, corresponds to 0,003 2 g of SO_2.
If the free sulphur dioxide content does not fall within the optimum range (0,18 to 0,31 g per 100 ml of reagent), adjust as appropriate, increasing the level by adding a calculated quantity of *di*sodium disulphite or decreasing it by bubbling air through the reagent solution.

3.4 *Methanol*, standard solution corresponding to 0,2 % (*V*/*V*) of methanol.

Place 2,00 ml of absolute methanol in a 1 000 ml one-mark volumetric flask, add a quantity of methanol-free ethanol corresponding to 98 ml of anhydrous ethanol, dilute to the mark with water and mix.

1 ml of this standard solution contains 0,002 ml of absolute 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 *Colorimetric tubes*, of capacity approximately 20 ml, fitted with ground glass stoppers.

4.2 *Water bath*, capable of being controlled at 20 ± 1 °C.

5 Procedure

5.1 Test portion and preparation of the test solution

Take as the test portion a volume (V_1) of the laboratory sample, corresponding to 10,0 ml of anhydrous ethanol, and place it in a 100 ml one-mark volumetric flask. Prepare the test portion by diluting to the mark with water and mixing. Transfer 5,0 ml of this solution to one of the colorimetric tubes (**4.1**).

5.2 *Preparation of standard solutions*, used for the preparation of standard colorimetric solutions

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

Into a series of five 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 10 % (*V*/*V*) solution of methanol-free ethanol in water and mix.

Standard methanol solution (3.4)	Corresponding volume of methanol
ml	ml
5,00	0,010
10,0	0,020
25,0	0,050
50,0	0,100
75,0	0,150

5.3 Preparation of standard colorimetric solutions

Into a series of five of the colorimetric tubes (**4.1**), place 5,0 ml of each of the standard methanol solutions (**5.2**).

Treat the contents of each of the tubes, including the tube containing 5,0 ml of the test solution (**5.1**), as follows.

Add 2,0 ml of the potassium permanganate solution (**3.1**), mix and allow to stand for 10 min in the water bath (**4.2**), controlled at 20 ± 1 °C. Then add 2,0 ml of the oxalic acid solution (**3.2**) and mix. At this stage, the solutions should be colourless and free from precipitated manganese. Add 5 ml of the Schiff reagent (**3.3**), mix and allow to stand for 1 h.

5.4 Determination

Examine the tubes vertically and note the standard colorimetric solution having a colour matching most closely that developed in the test solution.

NOTE In cases of doubt, choose the standard colorimetric solution having the lower concentration.

6 Expression of results

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

$$\frac{V_0 \times 100}{V_1}$$

where

V_0 is the volume, in millilitres, of methanol in the dilute standard solution (see **5.2**) from which was prepared the standard colorimetric solution having a colour matching most closely that developed in the test solution;

V_1 is the volume, in millilitres, of the test portion.

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