BRITISH STANDARD

BS 6392-4: 1983

Incorporating Amendment No. 1

Testing of ethanol for industrial use —

Part 4: Method for determination of aldehydes content

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 661.722:543.432.062:547.281

Foreword

This Part of BS 6392 is technically equivalent to ISO 1388 "*Ethanol for industrial use — Methods of test"* Part 5 *"Determination of aldehydes content — Visual colorimetric method*", published in 1981 by the International Organization for Standardization (ISO).

For ease of production, the text of ISO 1388-5:1981, 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 6392-4.

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
4940	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 aldehydes content of ethanol for industrial use.

The method is applicable to products having aldehyde contents, expressed as acetaldehyde, in the range 0,000 25 to 0,001 25 % (*m*/*m*).

NOTE The title of the publication referred to in this standard is given on inside back cover.

2 Principle

Reaction of the aldehydes present in a test portion with Schiff reagent. Visual comparison of the colour obtained with the colours of standard colorimetric solutions containing known quantities of acetaldehyde.

3 Reagents

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

3.1 *Ethanol,* 95 % (*V*/*V*), aldehydes-free, purified as follows.

Boil 1 500 ml of absolute ethanol, under reflux, for 2 h, with 15 g of *m*-phenylenediamine. Distil the mixture, rejecting the first and last 50 ml fractions of the distillate. Adjust the concentration to 95 % (*V*/*V*) by adding an appropriate volume of water and mix.

Use the procedure specified in clause **5** to verify that the purified ethanol is free from aldehydes.

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

WARNING — *m*-Phenylenediamine is toxic by inhalation, in contact with the skin and if swallowed. Avoid contact with skin and eyes, and inhalation of the dust.

3.2 *Schiff reagent*

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

3.2.1 *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 9,60 ± 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.

3.2.2 *Determination and adjustment of free sulphur dioxide content*

Transfer 10 ml of the colourless reagent (**3.2.1**) 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 it as appropriate, increasing the level by adding a calculated quantity of *di*sodium disulphite or decreasing it by bubbling air through the reagent solution.

3.3 *Acetaldehyde,* standard solution corresponding to 1 g of acetaldehyde per litre.

Weigh, to the nearest 0,000 1 g, 0,693 0 g of acetaldehyde ammonia [$CH_3CH(NH_2)OH$] and dissolve it in the ethanol (**3.1**). Transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark with ethanol of the same quality and mix.

1 ml of this standard solution contains 0,001 g of acetaldehyde.

NOTE 1 If acetaldehyde ammonia of analytical grade is not available, purify the commercial product as follows.
Dissolve about 5 g of acetaldehyde ammonia in a small quantity of absolute ethanol and precipitate it by adding twice the volume of dry diethyl ether ($C_2H_5OC_2H_5$). Using a Buchner funnel, filter the precipitate and, after washing it with more of the diethyl ether, transfer it immediately to a vacuum desiccator containing sulphuric acid, ϱ approximately 1,84 g/ml, 98 % (*m*/*m*) solution, as desiccant, and allow to dry for 3 to 4 h. Repeat the purification if necessary, until the product is colourless.

NOTE 2 Industrial methylated spirits 95 % (*V*/*V*) is not suitable for use in place of the absolute ethanol used in purifying the acetaldehyde ammonia.

WARNING — Diethyl ether is highly flammable and its vapour is harmful. Avoid breathing vapour.

3.4 *Acetaldehyde,* standard solution corresponding to 0,1 g of acetaldehyde per litre.

Transfer 25,0 ml of the standard acetaldehyde solution (**3.3**) to a 250 ml one-mark volumetric flask, dilute to the mark with the ethanol (**3.1**) and mix.

1 ml of this standard solution contains 0,000 1 g of acetaldehyde.

4 Apparatus

Ordinary laboratory apparatus, and

4.1 *Colorimetric tubes,* fitted with ground glass stoppers, of capacity about 20 ml, and graduated at 10 and 14 ml.

4.2 *Graduated pipettes,* of capacity 5 ml, graduated in 0,02 ml divisions.

5 Procedure

5.1 Test portion

Using one of the graduated pipettes (**4.2**), measure 3,0 ml of the laboratory sample into one of the colorimetric tubes (**4.1**).

5.2 Preparation of the test solution and standard colorimetric solutions

Into a series of six 100 ml one-mark volumetric flasks, place the volumes of the standard acetaldehyde solution (**3.4**) indicated in the following table, dilute to the mark with the ethanol (**3.1**) and mix.

Standard acetaldehyde solution (3.4)	Corresponding mass of acetaldehyde
ml	g
2,0	0,000 2
3,0	0,000 3
5,0	0,000 5
7,0	0,000 7
9,0	0,000 9
10,0	0,001 0

Using the graduated pipettes (**4.2**), measure, into a series of six of the colorimetric tubes (**4.1**), 3,0 ml of each of these diluted standard acetaldehyde solutions.

Treat the contents of each tube, including those of the tube containing the test portion (**5.1**), as follows.

Dilute to 10 ml with water and add sufficient of the Schiff reagent (**3.2**) to bring the volume to 14 ml. Stopper the tubes, mix the solutions (preferably simultaneously) and allow to stand in a rack for 25 min.

5.3 Determination

Compare the colour of the test solution with the colours of the standard colorimetric solutions, in dispersed daylight.

NOTE If the colour of the test solution is deeper than that of the most concentrated standard colorimetric solution, repeat the test using more of the laboratory sample suitably diluted with the ethanol (**3.1**), and take this into account in the calculation of results.

6 Expression of results

The aldehydes content, expressed as acetaldehyde (CH_3CHO) as a percentage by mass, is given by the formula

$$\frac{m}{\varrho}$$

where

m is the mass, in grams (see the table), of acetaldehyde used to prepare the diluted standard solution giving a colour matching most closely that developed in the test solution.

ϱ is the density, in grams per millilitre, of the sample at 20 °C (see BS 4522);

Publication referred to

BS 4522, *Method for the determination of density of liquids at 20 °C.*

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