BRITISH STANDARD

BS 6392-2: 1983

Incorporating Amendment No. 1

Testing of ethanol for industrial use —

Part 2: Method for determination of carbonyl compounds content present in small amounts (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 661.722:543.42.062:543.854.6

Foreword

This Part of BS 6392 is technically equivalent to ISO 1388 "*Ethanol for industrial use — Methods of test — Part 3: Estimation of content of carbonyl compounds present in small amounts — Photometric method*", published in 1981 by the International Organization for Standardization (ISO).

For ease of production, the text of ISO 1388-3:1981, 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 6392-2.

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 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
4938	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 estimation of the content of carbonyl compounds present in small amounts in ethanol for industrial use.

The method is applicable to products having carbonyl compounds contents, expressed as acetaldehyde, between 0,000 25 and 0,01 % (*m*/*m*).

NOTE 1 This method, which is used commercially, allows determination of only those carbonyl compounds which react under the specified conditions.
NOTE 2 The title of the publication referred to in this standard is given on inside back cover.

2 Principle

Reaction in acid medium of the carbonyl compounds in a test portion with 2,4-dinitrophenylhydrazine. Formation of the corresponding 2,4-dinitrophenylhydrazones, which, after making the solution alkaline, take on a red coloration.

Photometric measurement of this red coloration at a wavelength of about 445 nm.

3 Reagents

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

3.1 *Ethanol,* free from carbonyl compounds, purified as follows.

Boil under reflux 500 ml of ethanol with 5 g of 2,4-dinitrophenylhydrazine and 5 drops of the hydrochloric acid solution (**3.3**), for 2 to 3 h. Distil 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 coloured, redistil it.

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

3.2 *2,4-Dinitrophenylhydrazine,* saturated solution in the ethanol (**3.1**) at ambient temperature.

WARNING — 2,4-Dinitrophenylhydrazine is harmful by inhalation, in contact with the skin or if swallowed, and carries the risk of explosion by shock, friction, heat or other sources of ignition, particularly when dry. Avoid inhalation of dust. Avoid contact with skin and eyes. Take care in using the material, particularly if it is dry.

3.3 *Hydrochloric acid,* ϱ approximately 1,18 /ml, about 36 % (*m*/*m*) solution.

3.4 *Potassium hydroxide,* 100 g/l solution in a 70 % (*V*/*V*) solution of the ethanol (**3.1**).

3.5 *Carbonyl compounds,* standard solution corresponding to 0,440 g of carbonyl compounds, expressed as acetaldehyde, per litre.

Weigh, to the nearest 0,000 1 g, 1,200 g of acetophenone, and dissolve it in a little of the ethanol (**3.1**). Transfer quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark with ethanol of the same quality and mix. Take 10,0 ml of this solution, transfer it to a 100 ml one-mark volumetric flask, dilute to the mark with the ethanol (**3.1**) and mix.

1 ml of this standard solution contains 440 µg of carbonyl compounds, expressed as acetaldehyde.

4 Apparatus

Ordinary laboratory apparatus, and

4.1 *Water bath,* capable of being controlled at 50 ± 2 °C.

4.2 *Test tubes,* fitted with ground glass stoppers.

4.3 *Spectrophotometer,* or

4.4 *Photoelectric absorptiometer,* fitted with filters giving maximum transmission at a wavelength of about 445 nm.

5 Procedure

5.1 Test portion

Take 1,0 ml of the laboratory sample and place it in one of the test tubes (**4.2**).

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 1,0 ml of the ethanol (**3.1**).

5.3 Preparation of the calibration graph

5.3.1 Preparation of dilute standard solutions, with a view to preparation of standard colorimetric solutions

Into a series of seven 25 ml one-mark volumetric flasks, introduce the volumes of the standard carbonyl compounds solution (**3.5**) indicated in the following table and dilute to the mark with the ethanol (**3.1**).

Standard carbonyl compounds solution (3.5)	Corresponding mass of carbonyl compounds, expressed as CH_3CHO	Mass of carbonyl compounds in 1 ml of dilute standard solution
ml	µg	µg
0[a]	0	0
0,15	66,0	2,6
0,25	110,0	4,4
0,50	220,0	8,8
0,75	330,0	13,2
1,00	440,0	17,6
1,25	550,0	22,0

[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 seven of the test tubes (**4.2**), place 1,0 ml of each of the dilute standard solutions (**5.3.1**).

5.3.3 ***Colour development***

Add 1,0 ml of the 2,4-dinitrophenylhydrazine solution (**3.2**) and one drop of the hydrochloric acid solution (**3.3**). Stopper the tubes and heat for 30 min on the water bath (**4.1**), controlled at 50 ± 2 °C. Allow to cool, add 5,0 ml of the potassium hydroxide solution (**3.4**), mix, and allow to stand for 5 min.

5.3.4 ***Photometric measurements***

Immediately carry out the photometric measurements on each of the standard colorimetric solutions, using either the spectrophotometer (**4.3**), set at a wavelength of about 445 nm, or the photoelectric absorptiometer (**4.4**) fitted with appropriate filters, after having first adjusted the instrument to zero absorbance against the ethanol (**3.1**).

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 masses, in micrograms, of carbonyl compounds in 1 ml of each dilute standard solution (**5.3.1**) as abscissae, and the corresponding corrected values of absorbance as ordinates.

5.4 Determination

5.4.1 ***Colour development***

Treat the test portion (**5.1**) in the test tube, following the procedure specified in **5.3.3**.

5.4.2 ***Photometric measurements***

Immediately carry out the photometric measurements on the test solution and the blank test solution following the procedures specified in **5.3.4**, after having adjusted the instrument to zero absorbance against the ethanol (**3.1**).

NOTE If the absorbance exceeds the maximum of the calibration graph, repeat the determination (**5.4**) using as the test portion 1,0 ml of a test solution prepared by diluting 1,0 ml of the laboratory sample with an appropriate volume (not more than 4,0 ml) of the ethanol (**3.1**).

6 Expression of results

By means of the calibration graph (**5.3.5**), determine the masses of carbonyl compounds corresponding to the values of the photometric measurements.

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

$$\frac{(m_1 - m_0) \times 100}{1{,}0 \times \varrho \times 10^6} \times r_D$$

$$= \frac{m_1 - m_0}{\varrho \times 10^4} \times r_D$$

where

m_0 is the mass, in micrograms, of carbonyl compounds found in the blank test solution;

m_1 is the mass, in micrograms, of carbonyl compounds found in the test solution;

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

r_D is the ratio of the volume of the diluted test solution (see the note to **5.4.2**) to the volume of the aliquot portion taken for the determination (if the test portion was not diluted, r_D is equal to 1);

1,0 is the volume, in millilitres, of the test portion (**5.1**).

Publication referred to

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

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389 Chiswick High Road
London
W4 4AL