BRITISH STANDARD

BS 6392-3: 1983

Ethanol for industrial use —

Part 3: Method for determination of carbonyl compounds content present in moderate amounts (titrimetric 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 661.722:543.24.062:543.854.6

Foreword

This Part of BS 6392 is technically equivalent to ISO 1388 "*Ethanol for industrial use — Methods of test*" Part 4 "*Estimation of content of carbonyl compounds present in moderate amounts — Titrimetric method*", published in 1981 by the International Organization for Standardization (ISO).

For ease of production, the text of ISO 1388-4:1981, 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 6392-3.

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

Contents

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1 Scope and field of application

This Part of BS 6392 describes a titrimetric method for estimation of the content of carbonyl compounds present in moderate amounts in ethanol for industrial use.

The method is applicable to products having carbonyl compounds contents, expressed as acetaldehyde, equal to or greater than 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 the inside back cover.

2 Principle

Reaction of hydroxylammonium chloride with the carbonyl compounds in a test portion and titration of the hydrochloric acid formed with standard volumetric sodium hydroxide solution, in the presence of bromophenol blue as indicator.

3 Reagents

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

3.1 *Hydroxylammonium chloride reagent*

WARNING — Corrosive and irritating. Avoid contact with eyes and skin.

Dissolve 4 g of hydroxylammonium chloride in 20 ml of water and dilute to 200 ml with 95 % (*V*/*V*) ethanol. Heat under reflux for 30 min on a boiling water bath, cool to ambient temperature, add 5 ml of the bromophenol blue solution (**3.4**) and just sufficient of the sodium hydroxide solution (**3.2**) to produce a dichroic green coloration.

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

3.2 *Sodium hydroxide,* standard volumetric solution, c(NaOH) = 0,1 mol/l.

3.3 *Hydrochloric acid,* standard volumetric solution, c(HCl) = 0,1 mol/l.

3.4 *Bromophenol blue,* 2 g/l ethanolic solution.

Dissolve 0,2 g of bromophenol blue in 1,5 ml of the sodium hydroxide solution (**3.2**) and dilute to 100 ml with 95 % (*V*/*V*) ethanol.

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

4 Apparatus

Ordinary laboratory apparatus, and

4.1 *Conical flasks,* of capacity 150 ml, fitted with ground glass stoppers.

5 Procedure

5.1 Test portion

Take 50 ± 0,1 ml of the laboratory sample and place it in one of the conical flasks (**4.1**).

5.2 Determination

Place 50 ml of the hydroxylammonium chloride reagent (**3.1**) in a second conical flask (**4.1**), to be used as the colour standard.

Add 1,25 ml of the bromophenol blue solution (**3.4**) to the flask containing the test portion (**5.1**) and add, drop by drop, either the sodium hydroxide solution (**3.2**) or the hydrochloric acid solution (**3.3**) until the colour matches that of the colour standard. Then add, to each of the flasks, 25 ml of the hydroxylammonium chloride reagent (**3.1**) and stopper the flask containing the colour standard.

Loosely stopper the flask containing the test solution and heat it for 10 min on a boiling water bath. Remove the flask from the water bath, cool to ambient temperature and titrate the solution with the sodium hydroxide solution (**3.2**) until the colour matches as closely as possible that of the colour standard.

6 Expression of results

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

$$\frac{0{,}004\,405 \times V \times 100}{50 \times \varrho} = \frac{0{,}008\,81\,V}{\varrho}$$

where

V is the volume, in millilitres, of the sodium hydroxide solution (**3.2**) used for the determination;

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

0,004 405 is the mass, in grams, of carbonyl compounds, expressed as acetaldehyde, corresponding to 1 ml of sodium hydroxide solution, c(NaOH) = 0,100 mol/l;

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

NOTE If the concentrations of the standard volumetric solutions used are not exactly as specified in the list of reagents, an appropriate correction should be made.

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Publication referred to

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

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