BRITISH STANDARD

BS 6392-8: 1983

Incorporating Amendment No. 1

Testing of ethanol for industrial use —

Part 8: Method for determination of esters 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 661.722:543.851.1

Foreword

This Part of BS 6392 is technically equivalent to ISO 1388 "*Ethanol for industrial use — Methods of test*" Part 9 "*Determination of esters content — Titrimetric method after saponification*", published in 1981 by the International Organization for Standardization (ISO).

For ease of production, the text of ISO 1388-9:1981, 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 6392-8.

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
4944	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 titrimetric method, after saponification, for the determination of the esters content of ethanol for industrial use.

The method is applicable to products having esters contents, expressed as ethyl acetate, in the range 0,005 to 0,15 % (*m*/*m*).

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

2 Principle

Saponification of the esters present in a test portion by boiling with excess standard volumetric sodium hydroxide solution; determination of the residual sodium hydroxide, and hence the amount used for saponification, by titration with standard volumetric hydrochloric acid solution in the presence of phenolphthalein as indicator.

3 Reagents

During the analysis, unless otherwise specified, use only reagents of recognized analytical grade, and distilled water or water of equivalent purity, carbon dioxide-free, recently prepared.

3.1 *Water*, carbon dioxide-free.

Boil distilled water and allow it to cool in a flask fitted with a stopper carrying a soda-lime guard-tube.

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 *Phenolphthalein*, 5 g/l ethanolic solution.

Dissolve 0,5 g of phenolphthalein in 100 ml of 95 % (*V*/*V*) ethanol and add the sodium hydroxide solution (**3.2**) until a pale pink coloration is obtained.

4 Apparatus

Ordinary laboratory apparatus, and

4.1 *Conical flask,* of borosilicate glass, of capacity 500 ml, and having a ground neck.

4.2 *Reflux condenser*, water-cooled having a ground glass joint to fit the flask (**4.1**).

NOTE Clean the apparatus as follows.
Place a mixture of ethanol and sodium hydroxide solution (the proportions are not critical) in the flask (**4.1**), attach the reflux condenser (**4.2**) and heat the mixture gently under reflux for several minutes.
Discard the mixture and clean the flask and the condenser, first by washing with tap water and then by rinsing several times with distilled water.

5 Procedure

5.1 Test portion

Take 50,0 ml of the laboratory sample. If the esters content, expressed as ethyl acetate, is expected to be less than or equal to 0,01 % (*m*/*m*), take 100 ± 0,1 ml.

5.2 Determination

Place the test portion (**5.1**) in the flask (**4.1**), and add 20 ml of water and 0,5 ml of the phenolphthalein solution (**3.4**). Neutralize the solution [usually with the sodium hydroxide solution (**3.2**) until a pink coloration, persisting for about 15 s, appears].

Add 10,0 ml of the sodium hydroxide solution (**3.2**). Fit the condenser (**4.2**) and heat for 1 h on a boiling water bath. Fit a soda-lime guard-tube to the top of the condenser and cool the flask in water. Remove the soda-lime guard-tube and wash the inside of the condenser with two 10 ml portions of water, collecting the washings in the flask. Disconnect the flask and wash the ground neck with 10 ml of water, again collecting the washings in the flask.

Carefully titrate the solution with the standard volumetric hydrochloric acid solution (**3.3**) until the pink colour just disappears.

5.3 Blank test

Carry out a blank test on the neutral solution from the determination (**5.2**), using the procedure specified in **5.2**, paragraphs 2 and 3.

6 Expression of results

The esters content, expressed as a percentage by mass of ethyl acetate ($CH_3COOC_2H_5$), is given by the formula

$$\frac{0{,}008\,8 \times (V_2 - V_1) \times 100}{V_0\,\varrho}$$

$$= \frac{0{,}88 \times (V_2 - V_1)}{V_0\,\varrho}$$

where

V_0 is the volume, in millilitres, of the test portion (**5.1**);

V_1 is the volume, in millilitres, of the hydrochloric acid solution (**3.3**) used for the determination;

V_2 is the volume, in millilitres, of the hydrochloric acid solution (**3.3**) used for the blank test;

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

0,008 8 is the mass, in grams, of esters, expressed as ethyl acetate, corresponding to 1 ml of sodium hydroxide solution, c(NaOH) = 0,100 mol/l.

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.

Publication referred to

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

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