BRITISH STANDARD

BS 3762-3.24: 1988

Analysis of formulated detergents —

Part 3: Quantitative test methods —

Section 3.24 Methods for determination of low molecular mass alcohols content

NOTE It is recommended that this Section be read in conjunction with the information in the "*General introduction*", published separately as BS 3762-0.

Confirmed December 2011

UDC 661.185.6:[661.72 + 001.4]:543.620.1

Foreword

This Section of BS 3762 has been prepared under the direction of the Chemicals Standards Committee.

This standard describes methods of test only and should not be referred to as a specification defining limits of purity. Reference to the standard should indicate that the method of test used is in conformity with BS 3762-3.24.

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 to 4, 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 29 January 1988

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/34
Draft for comment 86/53319 DC

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Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

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1 Scope

This Section of BS 3762 describes two methods for the determination of low molecular mass alcohols in liquid detergent formulations.

Method 1 is intended for the determination of alcohols with a carbon chain of one to four and in a wide range of concentrations. Method 2 is for determining methanol and ethanol, from which their ratio can be calculated.

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

2 Principle

A known mass of acetonitrile (internal standard) in water is added to a known mass of sample and the mixture is subjected to analysis by packed column gas chromatography. The areas of the acetonitrile and alcohol peaks are obtained from an electronic integrator and the concentration of the alcohol(s) calculated using a response factor derived from calibration solutions.

If necessary, the alcohols may be identified by comparison of peak retention times.

3 Reagents

The reagents shall be of a recognized analytical grade. Water complying with grade 3 of BS 3978 shall be used throughout.

3.1 *Internal standard acetonitrile aqueous solution,* 75 mg/10 mL approximately.

3.2 *Samples of the appropriate alcohols*

4 Apparatus

Ordinary laboratory apparatus and the following are required.

4.1 *Gas chromatograph,* with flame ionization detector and capable of temperature programming, comprising a column capable of producing a chromatogram with resolution similar to, or better than, that shown in Figure 1 (e.g. 1.8 m × 2 mm internal diameter glass coil packed with porous polymer beads, 80 to 100 mesh).

NOTE Poropak Q has been found to be a suitable packing.

4.2 *Electronic integrator,* giving a linear response with adequate sensitivity, and satisfactory correction for deviation of the baseline.

4.3 *Syringe,* of 1 µL capacity, graduated in 0.1 µL.

4.4 *Safety pipette filler*

4.5 *Sample tubes,* 25 mL, fitted with screw caps with wadded aluminium inserts.

5 Instrument conditions

5.1 Temperature programme

The temperature programme shall be that required for the column (see **4.1**).

NOTE The following is given as an example:

initial temperature	170 °C
ramp rate	10 °C/min
final temperature	230 °C for 5 min
injector temperature	150 °C
detector temperature	250 °C

5.2 Gas flows

The carrier gas shall be oxygen-free nitrogen.

NOTE The instrument manual should be referred to for details of detector gases.

6 Procedure

6.1 Method 1

6.1.1 *Preparation of calibration data*

6.1.1.1 Pipette 10 mL of the internal standard acetonitrile solution (**3.1**) into each of three sample tubes (**4.5**) using the safety pipette filler (**4.4**).

6.1.1.2 Weigh, to the nearest 0.001 g, into each tube the following approximate quantities, in mg, of the appropriate alcohols.

	1	**2**	**3**
2-methylpropan-2-ol	75	50	100
2-methylpropan-1-ol	100	50	75
butan-2-ol	50	75	100
butan-1-ol	75	100	50
propan-2-ol	100	50	75
propan-1-ol	50	75	100
ethanol	75	100	50
methanol	50	75	100

Cap the tubes before each weighing and finally agitate to mix.

NOTE The above complex mixtures are for use if the type of alcohol(s) present is not known. If the type of alcohol(s) present is known, the mixtures may be simplified accordingly.

6.1.1.3 Inject approximately 0.7 µL of one of the calibration solutions (**6.1.1.2**) into the gas chromatograph (**4.1**) operating under the appropriate conditions (see clause **5**) and allow the chromatogram to develop.

6.1.1.4 Obtain from the electronic integrator (**4.2**) the peak areas of the acetonitrile and the appropriate alcohols.

6.1.1.5 Calculate the response factor for each alcohol using the following expression:

$$K_{ROH} = \frac{m_{AN} \times A_{ROH}}{m_{ROH} \times A_{AN}}$$

where

K_{ROH} is the response factor for the alcohol;

m_{AN} is the mass of the acetonitrile (in mg);

m_{ROH} is the mass of the alcohol (in mg);

A_{AN} is the peak area for the acetonitrile;

A_{ROH} is the peak area for the alcohol.

6.1.1.6 Proceed according to the method described in **6.1.1.3** to **6.1.1.5** for the other calibration solutions.

6.1.2 ***Procedure***

6.1.2.1 *Test portion.* Take a suitable laboratory sample and weigh, to the nearest 0.001 g, into a sample tube (**4.5**), a quantity which contains approximately 75 mg of an alcohol.

NOTE If more than one alcohol is present and the concentration of each is different, it is advisable to prepare new calibration data in which the standards will bracket each alcohol concentration in the sample.

6.1.2.2 *Determination.* Pipette 10 mL of the internal standard acetonitrile solution (**3.1**) into the tube using the safety pipette filler (**4.4**). Cap the tube and agitate to mix.

NOTE The same internal standard acetonitrile solution should be used for both calibration and analysis of sample.

Proceed according to the method described in **6.1.1.3** and **6.1.1.4**.

6.1.3 *Expression of results.* The content of each alcohol present in the sample, expressed as a percentage by mass, is given by the following expression:

$$\frac{m_{AN} \times A_{ROH}}{m_s \times A_{AN}} \times \frac{1 \times 100}{K_{ROH}}$$

where

m_{AN} is the mass of the acetonitrile (in mg);

m_s is the mass of the sample (in mg);

A_{ROH} is the peak area for the alcohol;

A_{AN} is the peak area for the acetonitrile;

K_{ROH} is the mean value of the response factor for the three calibration solutions.

6.2 Method 2

6.2.1 ***Preparation of calibration data***

6.2.1.1 Weigh, to the nearest 0.001 g, into a sample tube (**4.5**) approximately 1 g of methanol.

6.2.1.2 Weigh, to the nearest 0.001 g, into the sample tube approximately 9 g of water (thus obtaining an approximately 10 % (*m*/*m*) aqueous solution of methanol). Cap the tube and agitate to mix.

6.2.1.3 Pipette 10 mL of the internal standard acetonitrile solution (**3.1**) into a series of three samples tubes (**4.5**) using the safety pipette filler (**4.4**).

6.2.1.4 Weigh, to the nearest 0.001 g, into each tube the following approximate quantities in mg:

	1	**2**	**3**
methanol solution (see **6.2.1.2**)	50	30	25
ethanol	90	60	55

Cap the tubes before each weighing and agitate to mix.

6.2.1.5 Inject approximately 0.7 µL of one of the calibration solutions (**6.2.1.4**) into the gas chromatograph (**4.1**), operating under the appropriate conditions (see clause **5**) and allow the chromatogram to develop.

6.2.1.6 Obtain from the integrator (**4.2**) the peak areas of the acetonitrile, methanol and ethanol.

6.2.1.7 Calculate the response factor for each alcohol using the following expression.

$$K_{ROH} = \frac{m_{AN} \times A_{ROH}}{m_{ROH} \times A_{AN}}$$

where

K_{ROH} is the response factor for the alcohol;

m_{AN} is the mass of the acetonitrile used (in mg);

m_{ROH} is the mass of the alcohol (in mg);

A_{AN} is the peak area for the acetonitrile;

A_{ROH} is the peak area for the alcohol.

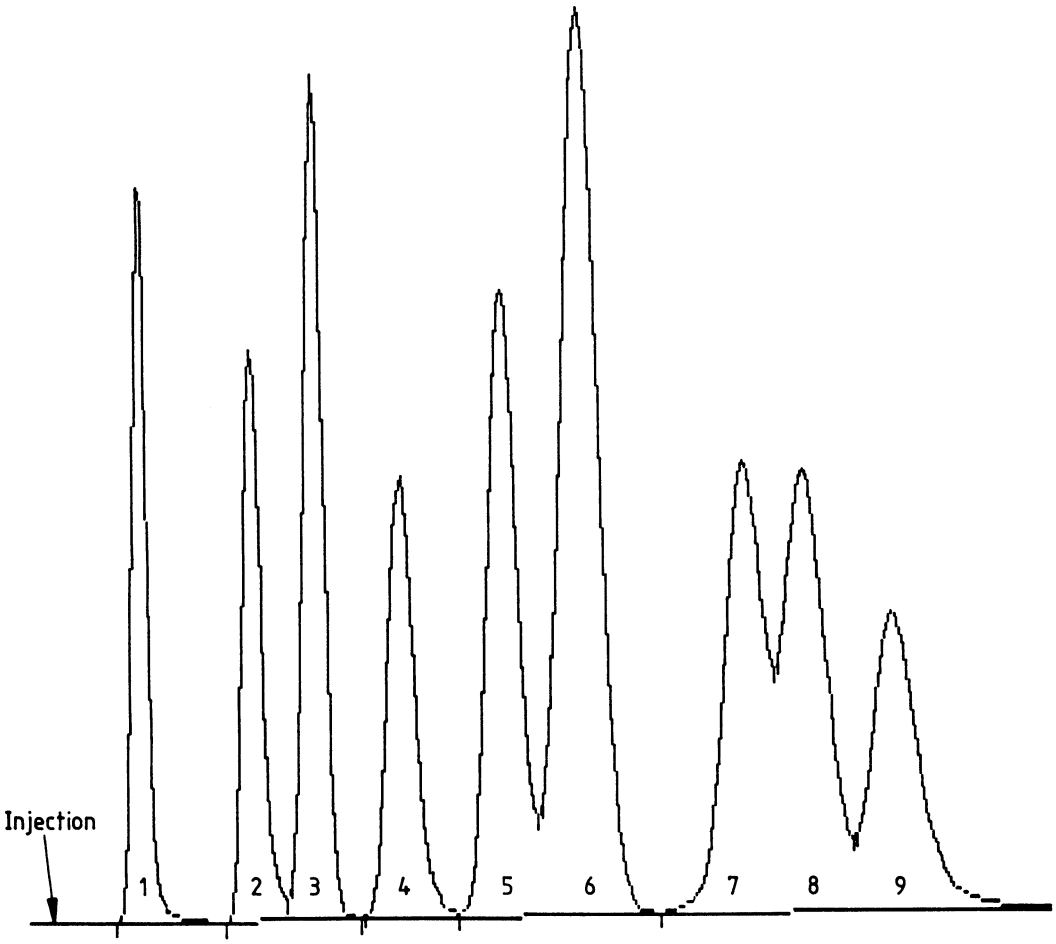
NOTE The mass of methanol used in this calculation is the calculated mass of pure methanol and not that of the 10 % solution.

6.2.1.8 Proceed according to the method described in **6.2.1.5** to **6.2.1.7** for the other calibration solutions.

6.2.2 ***Procedure***

6.2.2.1 *Test portion.* Take a suitable laboratory sample and weigh, to the nearest 0.001 g, into a sample tube (**4.5**) a quantity which contains approximately 70 mg of alcohol.

6.2.2.2 *Determination,* Pipette 10 mL of the internal standard acetonitrile solution (**3.1**) into the tube using the safety pipette filler (**4.4**). Cap the tube and agitate to mix.



Key

Peak No.	Relative retention to acetonitrile	Name
1	0.4	methanol
2	0.8	ethanol
3	1.0	acetonitrile
4	1.3	propan-2-ol
5	1.6	propan-1-ol
6	1.8	2-methylpropan-2-ol
7	2.4	butan-2-ol
8	2.6	2-methylpropan-1-ol
9	2.8	butan-1-ol

Figure 1 — Typical chromatogram

Proceed according to the method described in **6.2.1.5** and **6.2.1.6**.

6.2.3 *Expression of results.* The contents of each alcohol (methanol and ethanol) present in the sample, expressed as a percentage by mass, is given by the following expression.

$$\frac{m_{AN} \times A_{ROH}}{m_s \times A_{AN}} \times \frac{1 \times 100}{K_{ROH}}$$

where

m_{AN} is the mass of the acetonitrile (in mg);

m_s is the mass of the sample (in mg);

A_{ROH} is the peak area for the alcohol;

A_{AN} is the peak area for the acetonitrile;

K_{ROH} is the mean value of the response factor for the three calibration solutions.

Calculate the methanol/ethanol mass ratio.

7 Precision

No precision data are available.

8 Test report

The test report shall include the following information:

a) a reference to this British Standard, i.e. BS 3762-3.24:1988;

b) a reference to the test method used, i.e. method 1 or method 2;

c) the results expressed in accordance with **6.1.3** or **6.2.3**;

d) a complete identification of the sample.

Publication referred to

BS 3978, *Specification for water for laboratory use.*

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