BRITISH STANDARD

BS 3762-3.11: 1989

Analysis of formulated detergents —

Part 3: Quantitative test methods —

Section 3.11 Method for determination of free formaldehyde 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 January 2011

UDC 661.185:543.062:547.281.1

Foreword

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

This standard describes a method 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 3762-3.11.

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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 Policy Committee, was published under the authority of the Board of BSI and comes into effect on 31 January 1990

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 88/55447 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 a method for the determination of free formaldehyde in formulated detergents. It is not applicable if formaldehyde donors are present.

NOTE The titles of the publications referred to in this Section are listed on the inside back cover.

2 Principle

Reaction of the formaldehyde present with pentane-2,4-dione in the presence of ammonium acetate to form 3,5-diacetyl-1,4-dihydrolutidine. Spectrometric measurement of the absorbance of the resulting yellow complex at 410 nm.

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 *Pentane-2,4-dione reagent.* Dissolve 75 g of anhydrous ammonium acetate in approximately 200 mL of water. Add 1.0 mL of pentane-2,4-dione and 1.5 mL of glacial acetic acid. Dilute to 500 mL with water and mix well.

NOTE This reagent should be freshly prepared.

3.2 *Reference reagent.* Prepare as in **3.1** but without adding pentane-2,4-dione.

3.3 *Formaldehyde stock solution.* Accurately weigh, to the nearest 0.01 g, approximately 5 g of 370-400 g/L formaldehyde solution and transfer quantitatively to a 1 000 mL one-mark volumetric flask. Dilute to the mark with water and mix well.

Transfer 10.00 mL of this solution into a 250 mL Erlenmeyer flask, add 25.00 mL of iodine standard volumetric solution [c(I) = 0.100 mol/L] followed by 10 mL of sodium hydroxide solution [c(NaOH) = 1.0 mol/L]. Mix well and allow to stand for 5 min.

Add 11 mL of hydrochloric acid solution [c(HCl) = 1.0 mol/L] and titrate the excess iodine liberated with sodium thiosulphate standard volumetric solution [$c(Na_2S_2O_3)$ = 0.100 mol/L] using freshly prepared starch solution (5 g/L) as indicator. Record the volume, T, of sodium thiosulphate solution used, in mL.

NOTE 1.00 mL of 0.100 mol/L iodine solution is equivalent to 1.5 mg of formaldehyde.

3.4 *Formaldehyde working solution.* Transfer into a 100 mL one-mark volumetric flask 20.00 mL of the formaldehyde stock solution (**3.3**), dilute to the mark with water and mix well. Transfer into a 250 mL one-mark volumetric flask 5.00 mL of this solution, dilute to the mark with water and mix well.

1.00 mL of this solution contains approximately 8 µg of formaldehyde. Calculate the exact amount, expressed in µg/mL, using the expression

$$(25 - T) \times 0.6$$

where

T is the volume of sodium thiosulphate solution used (see **3.3**) (in mL).

3.5 *Propan-2-ol*

4 Apparatus

Ordinary laboratory apparatus and the following are required.

4.1 *Water bath,* capable of being controlled at 60 ± 1 °C.

4.2 *Spectrometer,* equipped with matched glass cells of optical path length 10 mm and capable of measuring absorbance at 410 nm.

4.3 *One-mark volumetric flasks,* 50 mL and 100 mL, complying with BS 1792.

5 Procedure

5.1 Calibration curve

Transfer into a series of the 50 mL one-mark volumetric flasks (**4.3**) 5.00 mL, 10.00 mL, 15.00 mL, 20.00 mL and 25.00 mL aliquot portions of the formaldehyde working solution (**3.4**). Add to each flask 15.00 mL of the pentane-2,4-dione reagent (**3.1**) and mix well.

Prepare a reagent blank by transferring into a 50 mL one-mark volumetric flask (**4.3**) 15.00 mL of water and 15.00 mL of the pentane-2,4-dione reagent (**3.1**).

Place the flasks in the water bath (**4.1**) controlled at 60 ± 1 °C for 10 min. Remove the flasks from the water bath, allow to cool to room temperature and dilute to the mark with the propan-2-ol (**3.5**). Mix well.

Using the spectrometer (**4.2**) measure the absorbance of each solution, including the reagent blank, in a cell of optical path length 10 mm at 410 nm, against water.

Subtract the absorbance of the reagent blank from the absorbance of each of the calibration solutions and construct a calibration curve of absorbance against the calculated number of micrograms of formaldehyde (see **3.4**).

NOTE Beer's law is not followed over this calibration range and hence the curve deviates slightly from linear.

5.2 Test portion

Weigh, to the nearest 0.001 g, a suitable quantity of the laboratory sample into a 250 mL beaker.

NOTE Table 1 may be used as a guide.

Table 1 — Guide to test portion size

Expected amount of formaldehyde	Mass of test portion
% *(m/m)*	g
0.1	1.0
0.05	2.5
0.025	5.0
0.01	10.0

5.3 Determination

Add 50 mL of water to the test portion (**5.2**) and stir to dissolve or disperse the test portion.

Transfer this solution quantitatively into a 100 mL one-mark volumetric flask (**4.3**) and dilute to the mark with water.

Transfer a 10.00 mL aliquot portion of this solution into a 50 mL one-mark volumetric flask (**4.3**), and add 15.00 mL of the pentane-2,4-dione reagent (**3.1**).

To eliminate interference due to any colour in the sample, use a reference solution, prepared as follows.

Transfer a second 10.00 mL aliquot portion into a 50 mL one-mark volumetric flask (**4.3**). Add 15.00 mL of the reference reagent (**3.2**).

Place the flasks in the water bath (**4.1**) controlled at 60 ± 1 °C for 10 min. Remove the flasks from the water bath, allow to cool to room temperature and dilute to the mark with the propan-2-ol (**3.5**). Mix well.

Using the spectrometer (**4.2**) measure the absorbance of the test solution in a cell of optical path length 10 mm at 410 nm, against the reference solution.

5.4 Blank solution

Prepare a reagent blank, as described in **5.1** and, using the spectrometer (**4.2**) measure its absorbance in a cell of optical path length 10 mm at 410 nm, against water.

6 Expression of results

Subtract the absorbance of the reagent blank from the absorbance of the test solution and read off from the calibration curve the amount, C, in micrograms, of formaldehyde in the test solution.

The formaldehyde content, expressed as a percentage by mass, is given by the expression:

$$\frac{C}{10^6} \times \frac{100}{10} \times \frac{100}{m}$$

$$= \frac{C}{1\,000 \times m}$$

where

m is the mass of the test portion (in g).

7 Repeatability

The difference found between the results of two determinations carried out on the same sample simultaneously or in rapid succession by the same analyst using the same apparatus should not exceed 2.5 % of the mean value.

8 Test report

The test report shall include the following information:

a) a reference to this British Standard, i.e. BS 3762-3.11:1989;

b) the results expressed in accordance with clause **6**;

c) a complete identification of the sample.

Publications referred to

BS 1792, *Specification for one-mark volumetric flasks.*
BS 3978, *Specification for water for laboratory use.*

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