BRITISH STANDARD

BS 3762-3.7: 1986

Analysis of formulated detergents —

Part 3: Quantitative test methods —

Section 3.7 Method for determination of total non-ionic matter 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 November 2008

UDC 661.185:543

Foreword

This Section of BS 3762 has been prepared under the direction of the Chemicals Standards Committee and supersedes method C1 of BS 3762:1964 in so far as it applies to formulated detergents, but that method will continue to exist until a British Standard for the determination in surface active agents (raw materials) has been published.

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 3762-3.7.

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.

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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 Committee, was published under the authority of the Board of BSI and comes into effect on
30 September 1986

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 85/52541 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 gravimetric method for the determination of the total non-ionic matter content of detergent products.

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

2 Principle

A suitable aliquot portion is taken from the ethanol soluble portion (see BS 3762-3.5).

The solution is stirred with a mixed bed of cation and anion exchange resins to remove ionic active matter. The solution is then filtered and evaporated, and the non-ionic matter is determined gravimetrically. This extract will include non-detergent organic matter and also any alkanolamide-type lather improver if present.

3 Reagents

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

3.1 *Ethanol*

NOTE For the purposes of **3.1**, the ethanol may be replaced by industrial methylated spirits complying with BS 3591, or such spirits diluted as required. It should be noted that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations, 1983 (S.I. 1983 No. 252). It is not permissible to use duty-free ethanol, received under the provisions of the Alcoholic Liquors Duties Act 1972, Section 10, for purposes for which industrial methylated spirits is an acceptable alternative.

3.2 *Acetone*

3.3 *A mixture of ion-exchange resins,* produced from a strongly acidic (H form) cation exchanger mixed with a strongly basic (OH form) anion exchanger in inverse proportion by mass approximating to their respective ion exchange capacities. Use 10 g of this mixed resin for products containing up to 4 % ethanol soluble ionic matter, and use an additional 5 g resin for each additional 4 % ethanol soluble ionic matter. Immediately before use wash the mixed bed resin, contained in a 250 mL conical flask, three times by swirling and decanting with twice its volume of ethanol (**3.1**) to remove water and any trace of ethanol-soluble contamination.

NOTE Ready prepared mixed bed resins are available commercially.

3.4 *Methylene blue solution,* 0.03 g/L.
Dissolve 0.15 g of methylene blue (CI 52015) in 100 mL of water, then dilute 2 mL of this solution to 100 mL.

3.5 *Chloroform*

4 Apparatus

Ordinary laboratory apparatus and the following are required.

4.1 *One-mark volumetric flask,* 250 mL, complying with BS 1792.

4.2 *Wide-mouthed, flat-bottomed flask,* 250 mL.

5 Procedure

5.1 Test portion

Carry out an ethanol extraction in accordance with BS 3762-3.5, by method 1 or method 2.
Pipette 100 mL of the solution from the one-mark volumetric flask (**4.1**) into the 250 mL conical flask containing the washed ion-exchange resin (**3.3**).

5.2 Determination

Insert a magnetic follower, cover the flask with a watch glass, and begin to stir, avoiding any splashing. Continue stirring for 20 min. Decant the solution into the 250 mL one-mark volumetric flask (**4.1**) through a 12.5 cm diameter filter paper (fast speed). Allow the solvent to drain thoroughly.

Add 20 mL of the ethanol (**3.1**) to the conical flask and swirl. Filter, allowing the solvent to drain completely. Repeat with three more 20 mL portions of ethanol. Wash the filter paper with ethanol. Dilute to the mark with ethanol.

Check the resin as follows. Remove exactly 5 mL of the solution from the volumetric flask using a clean, dry pipette and without rinsing the pipette with the solution. Transfer approximately 2 mL of this aliquot portion to a test tube and discard the rest. Add 10 mL of water, 1 mL of the methylene blue solution (**3.4**) and 1 mL of the chloroform (**3.5**) to the test tube, cover and shake. Allow the layers to separate. If the blue colour of the lower layer is more intense than that of the upper layer, the ion exchange was incomplete and the determination should be restarted using a fresh supply of ion-exchange resins; otherwise, continue with the determination.

NOTE 1 Ion-exchange resins have a finite exchange capacity and it is essential that they are not overloaded in terms of alcohol-soluble ionic constituents. It can arise that the exchange of anions is not complete.

Weigh, to the nearest 0.0005 g, a 250 mL wide-mouthed, flat-bottomed flask (**4.2**) containing a few anti-bumping granules. Transfer the solution quantitatively from the volumetric flask to this weighed flask.

Evaporate off the solvent on a steam bath to approximately 2 mL to 3 mL. Add 5 mL of the acetone (**3.2**) and evaporate to dryness while blowing with a gentle stream of air. Repeat with a further three portions of the acetone. Blow with a gentle stream of air until the last traces of acetone are removed. Wipe the outside of the flask dry. Cool in a desiccator for 30 min and weigh to the nearest 0.0005 g. Repeat the acetone drying procedure until two consecutive weighings agree within 0.002 g.

NOTE 2 The weighed residue may be used for the determination of alkanolamides content (see BS 3762-3.8).

6 Expression of results

The total non-ionic matter, expressed as a percentage by mass, is given by the following expression:

$$\frac{m_2 - m_1}{m_0} \times \frac{250}{245} \times \frac{250}{100} \times 100$$

where

m_0 is the mass of the sample taken in accordance with BS 3762-3.5 (in g);

m_1 is the mass of the evaporation flask (in g);

m_2 is the mass of the flask plus evaporated residue (in g).

NOTE The factor 250/245 is to correct for the 5 mL of solution removed in the check of the ion-exchange resin.

7 Precision

The precision is expected to depend markedly upon the content and the nature of the non-ionic matter. Some typical values are given below.

	Mean result	Repeatability	Reproducibility
	%	%	%
Detergent powder	1.5	0.27	0.43
Dishwashing liquid	8	0.44	1.2

The precision data were determined in an experiment conducted in 1985 involving seven laboratories.

NOTE For the meaning of the precision terms see BS 5497-1.

8 Test report

The test report shall include the following information:

a) a reference to this British Standard, i.e. BS 3762-3.7:1986;

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 3591, *Specification for industrial methylated spirits.*
BS 3762, *Analysis of formulated detergents.*
BS 3762-3.5, *Methods for determination of total organic matter content.*
BS 3762-3.8, *Method for determination of alkanolamides content.*
BS 3978, *Water for laboratory use.*
BS 5497, *Precision of test methods.*
BS 5497-1, *Guide for the determination of repeatability and reproducibility for a standard test method.*

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