BRITISH STANDARD

BS 3762-3.6: 1986

Analysis of formulated detergents —

Part 3: Quantitative test methods —

Section 3.6 Method for determination of matter soluble in light petroleum

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:[543.832.062:547.215/.217.2]

Foreword

This Section of BS 3762 has been prepared under the direction of the Chemicals Standards Committee and supersedes method B2 of BS 3762:1964, which is being deleted by amendment.

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 3762-3.6.

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 28 November 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 85/55067 DC

ISBN 0 580 15505 6

Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

blank

1 Scope

This Section of BS 3762 describes a method for the determination, in formulated detergents, of the matter soluble in light petroleum.

The matter soluble in light petroleum usually consists of unreacted fatty alcohols or hydrocarbons, but it may also contain added fatty matter and perfume.

Unreacted fatty matter from ingredients such as mono-glyceride sulphate or alkanolamide sulphate is not extracted with light petroleum, and ethoxylated alcohols, either added deliberately or present as unsulphated matter, are only partially extracted. These compounds can be determined, if desired, using the method described in BS 3762-3.7.

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

2 Principle

An aqueous alcoholic solution of the sample is extracted with light petroleum which is subsequently evaporated and the residue weighed. If the presence of soap is known or suspected, the solution is acidified before the extractions and the liberated fatty acids are subsequently washed out of the light petroleum extract by treatment with alkali and reserved for further examination if required.

3 Reagents

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

3.1 *Ethanol*

NOTE For the purposes of **3.1**, **3.2**, **3.3**, **3.4** and **3.5** 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 *Ethanol solution,* 70 % (*V*/*V*).

3.3 *Ethanol solution,* 50 % (*V*/*V*).

3.4 *Ethanol solution,* 40 % (*V*/*V*).

3.5 *Ethanol solution,* 25 % (*V*/*V*).

3.6 *Light petroleum,* boiling range 30 °C to 40 °C.

3.7 *Acetone*

3.8 *Sodium hydroxide solution,* c (NaOH) = 0.10 mol/L.

3.9 *Hydrochloric acid solution,* c (HCl) = 1.0 mol/L.

3.10 *Phenolphthalein indicator solution,* 5 g/L in 50 % ethanol solution.

4 Apparatus

Ordinary laboratory apparatus and the following are required.

4.1 *Separating funnels,* of 1 000 mL capacity.

4.2 *Flat-bottom flask,* of capacity not exceeding 250 mL.

4.3 *Steam bath*

5 Procedure

5.1 Test portion

If the sample is completely soluble in 50 % ethanol, weigh, to the nearest 0.01 g, sufficient sample to contain 1.6 g to 2.0 g of active matter, dissolve in the 50 % ethanol solution (**3.3**), transfer to one of the separating funnels (**4.1**) and dilute to 300 mL with the 50 % ethanol solution.

In other cases extract the organic matter, as described in BS 3762-3.5, up to the stage of diluting to 250 mL. If the ethanol soluble matter has been determined by method 1 of Section 3.5, transfer 50 mL of the ethanol solution thus obtained to a separating funnel (**4.1**) and add 250 mL of the 40 % ethanol solution (**3.4**). If method 2 of Section 3.5 has been used, transfer 100 mL of the ethanol solution thus obtained to a separating funnel and add 200 mL of the 25 % ethanol solution (**3.5**).

5.2 Determination

If the presence of soap is known or suspected, add the hydrochloric acid solution (**3.9**) until the pH is just below **3.5** (for example, as shown by indicator papers).

Add 100 mL of the light petroleum (**3.6**), swirl gently but thoroughly to give adequate extraction and allow the two phases to separate, adding a few millilitres of the ethanol (**3.1**) when necessary to break tenacious emulsions. Run off the aqueous ethanolic phase into a second separating funnel (**4.1**) and extract with a further 100 mL of the light petroleum.

Repeat the extraction of the aqueous ethanolic phase in a third separating funnel (**4.1**) with a further 100 mL of the light petroleum. Combine the three extracts in the first separating funnel and wash six times with fresh 50 mL quantities of the 70 % ethanol solution (**3.2**).

NOTE 1 If auxiliary surface active agents (alkanolamide foam stabilizers) and sarcosinates are known to be absent, the washing procedure may be simplified. Give four washes with 50 % ethanol solution (**3.3**) instead of six washes with 70 % ethanol solution.

Discard the aqueous ethanolic phase and washings.

Wash the light petroleum extract alternately three times each with 25 mL of the sodium hydroxide solution (**3.8**) and water, and finally with water until the wash liquor is no longer alkaline to the phenolphthalein indicator solution (**3.10**).

NOTE 2 If soap and sarcosinates are known to be absent, the alkali washes can be omitted.

Free fatty acids when present will be included in this fraction.

Transfer the washed light petroleum extract in stages to the flask (**4.2**), previously weighed to the nearest 0.001 g, and evaporate off the solvent.

WARNING. Appropriate precautions should be taken when evaporating the solvent as it is highly flammable.

Add 10 mL of the acetone (**3.7**) and again evaporate off the solvent, rotating the flask on the steam bath (**4.3**) during this operation to remove as much of the solvent as possible. Cool the flask to about 40 °C, gently blow off the last traces of the solvent with a current of dry air, cool and weigh to the nearest 0.001 g.

6 Expression of results

The matter soluble in light petroleum, expressed as a percentage by mass, is given by the following expression:

$$\frac{100 \times m_1}{m_2}$$

where

m_1 is the mass of extract (in g);

m_2 is the mass of sample taken or of sample represented by the volume of ethanol extract taken (in g).

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 3762-3.6:1986;

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

c) a complete identification of the sample;

d) any operational details regarded as optional.

Publications referred to

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 3762-3.7, *Method for determination of total non-ionic matter content.*
BS 3978, *Specification for water for laboratory use.*

BSI — British Standards Institution

BSI is the independent national body responsible for preparing British Standards. It presents the UK view on standards in Europe and at the international level. It is incorporated by Royal Charter.

Revisions

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions.

It is the constant aim of BSI to improve the quality of our products and services. We would be grateful if anyone finding an inaccuracy or ambiguity while using this British Standard would inform the Secretary of the technical committee responsible, the identity of which can be found on the inside front cover. Tel: 020 8996 9000. Fax: 020 8996 7400.

BSI offers members an individual updating service called PLUS which ensures that subscribers automatically receive the latest editions of standards.

Buying standards

Orders for all BSI, international and foreign standards publications should be addressed to Customer Services. Tel: 020 8996 9001. Fax: 020 8996 7001.

In response to orders for international standards, it is BSI policy to supply the BSI implementation of those that have been published as British Standards, unless otherwise requested.

Information on standards

BSI provides a wide range of information on national, European and international standards through its Library and its Technical Help to Exporters Service. Various BSI electronic information services are also available which give details on all its products and services. Contact the Information Centre. Tel: 020 8996 7111. Fax: 020 8996 7048.

Subscribing members of BSI are kept up to date with standards developments and receive substantial discounts on the purchase price of standards. For details of these and other benefits contact Membership Administration. Tel: 020 8996 7002. Fax: 020 8996 7001.

Copyright

BSI
389 Chiswick High Road
London
W4 4AL