BRITISH STANDARD

BS 3762-3.9: 1986

Analysis of formulated detergents —

Part 3: Quantitative test methods —

Section 3.9 Method for determination of soap 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 2009

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Foreword

This Section of BS 3762 has been prepared under the direction of the Chemicals Standards Committee and supersedes method B12 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 3762-3.9.

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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 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 85/55068 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 the soap content of formulated detergents. The mean relative molecular mass of the soap has to be known.

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

2 Principle

An acidified solution of the detergent is extracted with light petroleum. The extract is concentrated by evaporation and titrated with sodium hydroxide standard volumetric solution.

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** 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 *Light petroleum*, boiling range 40 °C to 60 °C.

3.3 *Sulphuric acid solution,* $c(H_2SO_4)$ = 5 mol/L approximately.

3.4 *Sodium hydroxide standard volumetric solution,* c(NaOH) = 0.100 mol/L.

3.5 *Bromophenol blue indicator solution*

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

3.7 *Sodium chloride*

4 Apparatus

Ordinary laboratory apparatus and the following are required.

4.1 *Separating funnels*, of 250 mL capacity.

4.2 *Flask*, of 250 mL capacity, fitted with a 24/29 ground glass joint (see BS 572).

4.3 *Distillation apparatus*, to fit **4.2**.

5 Procedure

5.1 Test portion

Weigh, to the nearest 0.01 g, approximately 5 g of synthetic detergent or approximately 3 g of formulated soap-based powder into a 150 mL beaker.

5.2 Determination

5.2.1 Add to the test portion 50 mL of water, heat to between 40 °C and 50 °C, stir and add 50 mL of the ethanol (**3.1**) and a few drops of the bromophenol blue indicator solution (**3.5**).

5.2.2 Add the sulphuric acid solution (**3.3**) drop by drop until the solution is acid. Transfer the solution to a separating funnel (**4.1**). Wash the beaker with 50 mL of the light petroleum (**3.2**) and transfer the washings to the separating funnel.

5.2.3 Stopper the separating funnel (**4.1**) and shake vigorously, occasionally inverting the separating funnel and opening the stopcock to release the pressure. Allow the layers to separate.

NOTE If difficulty is experienced in obtaining a satisfactory separation of the layers, the addition of small amounts of sodium chloride (**3.7**) or warming of the mixture may assist.

5.2.4 Remove the stopper and run the lower layer into a second separating funnel (**4.1**). To this second separating funnel add 50 mL of the light petroleum (**3.2**).

5.2.5 Repeat the procedure in **5.2.3**, then remove the stopper, run the lower layer into the original beaker and transfer the petroleum layer to the separating funnel containing the first extract.

5.2.6 Extract the aqueous layer with a further 50 mL of the light petroleum (**3.2**) using the second separating funnel and transfer the petroleum layer to the separating funnel containing the first two extracts. Wash once with 50 mL of water and discard the aqueous layer.

5.2.7 Transfer quantitatively the combined petroleum extracts to the flask (**4.2**). Evaporate to approximately 5 mL using the distillation apparatus (**4.3**).

5.2.8 Add to the flask (**4.2**) 100 mL of hot ethanol (**3.1**) neutralized to phenolphthalein and titrate with the sodium hydroxide solution (**3.4**) using the phenolphthalein indicator solution (**3.6**).

6 Expression of results

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

$$V \times 0.1 \times \frac{M}{1000} \times \frac{100}{m}$$

$$= \frac{V \times M \times 0.01}{m}$$

where

V is the volume of the sodium hydroxide solution required (in mL);

M is the mean relative molecular mass of the soap;

m is the mass of the test portion (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 3762-3.9:1986;

b) the results expressed in accordance with clause **6**, including the relative molecular mass used in the calculation;

c) a complete identification of the sample.

Publications referred to

BS 572, *Specification for interchangeable conical ground glass joints.*
BS 3591, *Specification for industrial methylated spirits.*
BS 3978, *Specification for water for laboratory use.*

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