BRITISH STANDARD

BS 3762-3.5: 1986

Analysis of formulated detergents —

Part 3: Quantitative test methods —

Section 3.5 Methods for determination of total organic 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 B1 of BS 3762:1964, which has been 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 3762-3.5.

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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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 85/52540 DC

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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.

Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

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1 Scope

This Section of BS 3762 describes methods for separating organic constituents from most of the inorganic constituents, and for determining the total organic matter content of formulated detergents.

Two procedures are described. Method 1 is widely applicable and is intended mainly for solid samples. Method 2 is restricted to samples containing less than 10 % alcohol-insoluble matter and is intended mainly for liquids.

Small but significant amounts of alkali metal carbonates, hydroxides and chlorides may accompany the organic matter, and organic compounds that are volatile, e.g. perfumes and unsulphonated alkylates, or that are sparingly soluble in ethanol, will not be completely determined.

Consequently, the gravimetric part of the method is applicable only when the proportions of these classes of compounds are negligible or when an approximate indication of the total organic matter content is adequate.

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

2 Principle

Extraction with hot ethanol, filtration and, if a quantitative result is required, evaporation of an aliquot portion of the filtrate and weighing the residue.

3 Reagents

The reagents shall be of a recognized analytical reagent grade.

3.1 *Ethanol*

NOTE For the purposes of **3.1**, the ethanol may be replaced by industrial methylated spirits complying with BS 3591, of 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*

4 Apparatus

Ordinary laboratory apparatus and the following are required.

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

5 Procedure

5.1 Method 1

5.1.1 *Test portion*. Weigh, to the nearest 0.01 g, about 10 g of the laboratory sample into a 600 mL beaker.

5.1.2 *Extraction of organic matter*. Add approximately 250 mL of the ethanol (**3.1**) to the test portion.

Cover with a watch glass, heat and stir with a mechanical or magnetic stirrer until the ethanol is boiling. Continue boiling and stirring for 5 min. Allow the beaker and contents to cool to about 60 °C and the insoluble matter to settle. Filter the ethanolic phase through a medium grade filter paper. Repeat this extraction twice more with new 250 mL portions of ethanol.

Add approximately 75 mL of the ethanol at 50 °C to 60 °C to the beaker containing the insoluble matter; break any remaining hard lumps with a glass rod. Allow the insoluble matter to settle and filter through the same filter paper.

Repeat this operation twice more.

Evaporate the combined filtrate and washings to less than 250 mL. Transfer to the 250 mL one-mark volumetric flask (**4.1**), cool and dilute to the mark with ethanol.

NOTE A portion of the ethanol soluble fraction may be used for the determination of total non-ionic matter content (see BS 3762-3.7).

5.1.3 *Determination*. Shake to disperse any precipitate, and pipette 100 mL of the alcoholic solution into a tared 250 mL beaker.

Evaporate on a steam bath in a fume cupboard. Inspect occasionally and guard against loss of sample by excessive foaming or bumping.

Add 10 mL of the acetone (**3.2**), and evaporate to dryness. Inspect occasionally and guard against loss of sample by excessive foaming or bumping. Allow to cool in a desiccator and weigh.

Repeat the operations of the previous paragraph until successive weighings agree to within 0.003 g.

5.2 Method 2

5.2.1 *Test portion*. Weigh, to the nearest 0.001 g, about 4 g of the laboratory sample into a 600 mL beaker.

5.2.2 *Extraction of organic matter*. Add about 100 mL of the ethanol (**3.1**) to the beaker. Cover with a watch glass and commence stirring and heating on a steam bath in a fume cupboard until the alcohol is boiling. Continue boiling for 5 min. Allow to cool to about 60 °C and filter through a medium fast filter paper (fine grade for liquid scourers) into the 250 mL one-mark volumetric flask (**4.1**).

Wash the beaker and residue in the filter paper with about 50 mL of hot ethanol. Repeat once. Allow to cool. Dilute to the mark with cold ethanol and mix.

5.2.3 *Determination*. Proceed in accordance with **5.1.3**.

6 Expression of results

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

$$m_1 \times \frac{250}{100} \times \frac{100}{m_2} = \frac{m_1 \times 250}{m_2}$$

where

m_1 is the mass of residue (in g);

m_2 is the mass of sample taken (in g).

NOTE The results should be interpreted with caution according to the requirements of the determination (see clause **1**).

7 Precision

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

	Mean result	Repeatability	Reproducibility
	%	%	%
Detergent powder (Method 1)	14	1.0	2.2
Dishwashing liquid (Method 2)	42	1.2	3.8

The precision data were determined from 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 3762-3.5:1986;

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

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

d) 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 3762-3.7, *Method for determination of total non-ionic matter content.*
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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