BRITISH STANDARD

BS 3762-2: 1989

Analysis of formulated detergents —

Part 2: Qualitative test methods

NOTE It is recommended that this Part be read in conjunction with the information in the "*General introduction*", published separately as BS 3762-0.

Confirmed January 2011

UDC 661.185.6:620.1

Foreword

This Part of BS 3762 has been prepared under the direction of the Chemicals Standards Committee and supersedes Part 2: Section A and method D7.A of BS 3762:1964, which are being deleted by amendment.

This standard describes methods 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 3762-2.

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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 February 1989

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 87/51452 DC

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Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

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1 Scope

This Part of BS 3762 describes qualitative tests which may be required as preliminary steps to the application of methods of analysis described in other Parts and Sections of BS 3762. The tests are for type of surface active agent, oxidizing agents, peroxy-compounds, urea and fluorescent whitening agents.

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

2 Reagents

The reagents shall be of a recognized analytical grade. Water complying with grade 3 of BS 3978 shall be used throughout.

2.1 *Chloroform*

2.2 *Ammonium hexathiocyanatocobaltate solution.* Dissolve 20 g of ammonium thiocyanate and 3 g of cobalt nitrate in about 70 mL of water and dilute to 100 mL.

2.3 *p-dimethylaminobenzaldehyde solution.* Dissolve 1 g of *p*-dimethylaminobenzaldehyde in 100 mL of ethanol[1)] plus 0.2 mL of hydrochloric acid solution [36 % (*m*/*m*)].

2.4 *Mixed indicator*

2.4.1 *Dimidium bromide/acid blue 1 stock solution.* This solution is prepared from acid blue 1 (CI 42045) (sodium α-[4-(diethylamino) phenyl] -α-[4-(diethylimino) cyclohexa-2,5-dienylidene] toluene-2,4-disulphonate) and dimidium bromide (3,8-diamino-5-methyl-6-phenylphenanthridinium bromide).

NOTE This solution is available commercially under the name "dimidium bromide-disulphine blue indicator stock solution".

Weigh 0.5 ± 0.005 g of dimidium bromide into a 50 mL beaker and 0.25 ± 0.005 g of acid blue 1 into a second 50 mL beaker. Add between 20 mL and 30 mL of hot 10 % (*V*/*V*) ethanol[1)] to each beaker. Stir until dissolved and transfer the solutions to a 250 mL one-mark volumetric flask with the ethanol solution and dilute to the mark with 10 % (*V*/*V*) ethanol[1)].

2.4.2 *Mixed acid indicator solution.* Add 200 mL of water to 20 mL of the stock solution (**2.4.1**) in a 500 mL one-mark volumetric flask. Add 20 mL of the sulphuric acid solution (**2.8**), mix and dilute to the mark with water. Store out of direct sunlight.

2.5 *Potassium iodide solution, c* (KI) = 0.5 mol/L.

2.6 *Potassium titanium oxalate solution,* 5 g/L.

2.7 *Sodium hydroxide solution, c* (NaOH) = 5 mol/L.

2.8 *Sulphuric acid solution, c* (H_2SO_4) = 2.5 mol/L.

3 Procedure

3.1 Preparation of test solution

Mix approximately 5 g of the sample with 95 mL of water until all soluble components appear to have been dissolved. Filter to remove any insoluble matter.

NOTE It is not necessary for the filtrate to be clear, as an emulsion, which will usually disappear, will not interfere with subsequent observations.

3.2 Test for type of surface active agent (acid medium)

Mix one drop of the test solution (**3.1**), 5 mL of the mixed acid indicator solution (**2.4.2**) and 5 mL of the chloroform (**2.1**). Allow the layers to separate. If the chloroform layer is not distinctly blue or pink, add a few more drops of the test solution, shake and allow to separate. Repeat if necessary until about 1 mL of the test solution has been added.

A pink colour in the chloroform layer denotes the presence of a synthetic anionic-active surface-active agent. A blue colour in the chloroform layer denotes the presence of a long chain amine, long chain amine oxide, or cationicactive or ampholytic surface-active agent. In the case of a blue colour or no colour, proceed in accordance with **3.3**.

3.3 Test for type of surface active agent (alkaline medium)

Mix one drop of the test solution (**3.1**), 5 mL of the mixed acid indicator solution (**2.4.2**), 1 mL of the sodium hydroxide solution (**2.7**) and 5 mL of the chloroform (**2.1**). Allow the layers to separate. If the chloroform layer is not distinctly blue or pink, add a few more drops of the test solution, shake and allow to separate. Repeat if necessary until about 1 mL of the test solution has been added.

A pink colour in the chloroform layer denotes the presence of a soap or synthetic anionic-active or ampholytic surface-active agent. Use the results from **3.2** to distinguish these agents. A blue colour in the chloroform layer denotes the presence of a cationic-active surface-active agent.

[1)] The ethanol may be replaced by industrial methylated spirits complying with BS 3591, or such spirits diluted as required. The use of industrial methylated spirits is governed by the Methylated Spirits Regulations, 1983 (SI 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.

3.4 Test for non-ionic surface-active agents

Mix 1 mL of the test solution (**3.1**), 10 mL of the ammonium hexathiocyanatocobaltate solution (**2.2**) and 5 mL of the chloroform (**2.1**). Allow the layers to separate.

A blue colour in the chloroform layer (in case of doubt, compare with a blank test, i.e. omitting the test solution) denotes the presence of a non-ionic surface-active agent.

NOTE Non-ionic surface active agents that contain less than three ethylene oxide units per molecule, including those not based on ethylene oxide, will not be detected by this test.

3.5 Test for oxidizing agents

Mix 5 mL of the test solution (**3.1**) with a few drops of the sulphuric acid solution (**2.8**) and add 1 mL of the potassium iodide solution (**2.5**).

A yellow or yellow-brown colour indicates the presence of an oxidizing agent.

3.6 Test for peroxy-compounds

Mix 5 mL of the test solution (**3.1**) with 1 mL of the sulphuric acid solution (**2.8**) and add 1 mL of the potassium titanium oxalate solution (**2.6**).

A yellow or orange colour indicates the presence of a peroxy-compound. A negative reaction in conjunction with a positive reaction in **3.4** suggests the presence of a chlorine bleach.

3.7 Test for urea

Place a drop of the test solution (**3.1**) on to a filter paper and add a drop of the *p*-dimethylaminobenzaldehyde solution (**2.3**). Hang the paper up to dry.

A bright yellow spot indicates the presence of urea or certain amines or other amides; absence of a yellow spot indicates the absence of urea.

3.8 Test for fluorescent whitening agents

Dissolve or disperse 2 g of the sample in 100 mL of water. Take two strips of filter paper of size approximately 5 cm × 2 cm and dip one to a depth of 2 cm to 3 cm in the solution and dip the other into distilled water. Remove the strips and compare them under a source of ultraviolet light, preferably in a darkened room.

Note whether the test strip shines more brightly than the control, indicating the presence of a fluorescent agent.

4 Test report

The test report shall include the following information:

a) a reference to this British Standard, i.e. BS 3762-2:1989;

b) a reference to the test method(s) used;

c) the result(s);

d) a complete identification of the sample.

Publications referred to

BS 3591, *Specification for industrial methylated spirits.*
BS 3978, *Specification for water for laboratory use.*

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