

Analysis of formulated detergents —

Part 3: Quantitative test methods —

Section 3.12 Method for determination of ammonia 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.

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Foreword

This Section of BS 3762 has been prepared under the direction of the Chemicals Standards Committee and supersedes method D1 of BS 3762:1964. **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 3762-3.12.**

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The committees responsible for this British Standard are shown in Part 0. The following BSI references relate to the work on this standard:
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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

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1 Scope

This Section of BS 3762 describes a method of analysis for the determination of ammonia content of formulated detergents.

This method is unsuitable for detergents containing urea.

A variation of the method is described for use when the detergent contains monoalkanolamines.

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

2 Principle

The ammonia present is determined by direct distillation from an alkaline solution of the detergent and titration of the distillate collected in boric acid solution. When monoalkanolamines are present, a modified procedure is used.

3 Reagents

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

3.1 Zinc dust

3.2 Oleyl alcohol, technical

3.3 Silicone ester, liquid

3.4 Boric acid 20 g/L solution. Dissolve 10 g of boric acid in water, warm if necessary to aid dissolution and dilute to 500 mL.

3.5 Sodium hydroxide solution, $c(\text{NaOH}) = 8 \text{ mol/L}$ approximately.

3.6 Hydrochloric acid solution, $c(\text{HCl}) = 0.100 \text{ mol/L}$.

3.7 Mixed indicator solution, comprising:

- a) 0.05 % (m/V) methyl red in 95 % (V/V) ethanol, and;
- b) 0.15 % (m/V) methylene blue solution.

Mix four parts of a) with one part of b). Prepare freshly each day.

3.8 Phenolphthalein indicator, 0.5 % (m/V) solution in 50 % (V/V) ethanol.

NOTE For the purposes of 3.7 and 3.8, the ethanol may be replaced by industrial methylated spirits 95 % (V/V) 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.

4 Apparatus

Ordinary laboratory apparatus and the following are required.

4.1 Distillation apparatus (for general method), Kjeldahl type, capacity not less than 300 mL.

NOTE The distillation apparatus may be fitted with a device to prevent foaming.

4.2 Distillation apparatus (for modified method, used when monoethanolamine is present), comprising:

- a) *distillation flask*, capacity 250 mL, three necked. In the side necks are fitted, respectively, a thermometer pocket and a tap funnel. In the centre neck is fitted a reflux condenser, and;
- b) *receiver, Drechsel bottle*, capacity 250 mL, with its inlet connected to the top of the reflux condenser and its outlet to a filter pump.

5 Procedure

5.1 Test portion

Weigh, to the nearest 0.01 g, an appropriate amount of the well mixed sample (usually 1 g to 5 g) into the distillation flask (4.1 or 4.2).

5.2 Determination

5.2.1 General method. Add 100 mL of water, a pinch of zinc dust (3.1) and about 5 mL of oleyl alcohol (3.2) or a few drops of silicone ester to the flask and then connect it to the tap funnel and stillhead. In the receiver place 50 mL of the boric acid solution (3.4) and about five drops of the mixed indicator solution (3.7).

By means of the tap funnel, add a few drops of the phenolphthalein indicator to the distillation flask, followed by sufficient of the sodium hydroxide solution (3.5) to render the contents alkaline and a few millilitres in excess. Keep the tap of the funnel closed except when reagents are actually being added.

Heat the contents of the distillation flask to the boil, and boil until between half and two-thirds of the water present has distilled into the receiver. Wash the inner surface of the condenser and the tip with water, collecting the washings in the receiver. Titrate the contents of the receiver with the hydrochloric acid solution (3.6). The colour changes from green to grey, and then to purple with excess acid.

Prepare a blank containing the same volumes of the boric acid solution, indicator, and water as for the test titration, and titrate to the same coloured end point as the test solution.

5.2.2 Modified method (for use when monoalkanolamines are present). Proceed as in the general method (see **5.2.1**) but, instead of distilling the ammonia solution by boiling, heat the contents of the distillation flask to 100 °C and maintain at this temperature for 1 h while passing a slow stream of air through the apparatus.

6 Expression of results

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

$$\frac{(V_1 - V_2) \times c \times 0.017 \times 100}{m}$$

where

- V_1 is the volume of the hydrochloric acid solution (**3.6**) used for the test solution (in mL);
- V_2 is the volume of the hydrochloric acid solution (**3.6**) used for the blank (in mL);
- c is the concentration of the hydrochloric acid solution (**3.6**) (in mol/L);
- m is the mass of the test portion (in g).

7 Test report

The test report shall include the following information:

- a) a reference to this British Standard, i.e. BS 3762-3.14;
- b) the results and the method of expression used;
- c) the test conditions.

Publications referred to

BS 3591, *Industrial methylated spirits*.

BS 3978, *Water for laboratory use*.

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