BRITISH STANDARD

BS 3762-3.8: 1986

Analysis of formulated detergents —

Part 3: Quantitative test methods —

Section 3.8 Method for determination of alkanolamides 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 D8 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 3762-3.8.

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.

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 85/52542 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 a method of analysis for the determination of the alkanolamides content of formulated detergents. The result is expressed as percentage alkanolamides, calculated using a stated relative molecular mass.

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

2 Principle

An aliquot portion of a solution of the non-ionic components, obtained in accordance with BS 3762-3.7, is evaporated and then heated with sulphuric acid, sodium sulphate and selenium to convert the alkanolamides to ammonium sulphate. Ammonia is subsequently distilled off and determined by titration with hydrochloric acid.

NOTE This is a form of the Kjeldahl method.

3 Reagents

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

3.1 *Ethanol*

NOTE For the purposes of **3.1** and **3.8**, 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 *Selenium*

3.3 *Sodium sulphate*, anhydrous.

3.4 *Sulphuric acid*, concentrated, (ρ = 1.84 g/mL).

3.5 *Orthoboric acid (boric acid)*, 20 g/L *solution*. Dissolve 10 g of orthoboric acid in water, warm if necessary to aid dissolution and dilute to 500 mL.

3.6 *Sodium hydroxide solution,* c (NaOH) = 10 mol/L approximately.

3.7 *Hydrochloric acid, standard volumetric solution,* c (HCl) = 0.100 mol/L.

3.8 *Mixed indicator solution*, comprising:

a) 0.5 g/L methyl red in 95 % (*V*/*V*) ethanol;

b) 1.5 g/L methylene blue solution.

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

4 Apparatus

Ordinary laboratory apparatus and the following are required.

4.1 *One-mark volumetric flask*, 100 mL, complying with BS 1792.

4.2 *Kjeldahl flasks*, 50 mL.

4.3 *Distillation apparatus*

For steam distillation (**5.3.1**) use **4.3.1** and **4.3.2**.

For direct distillation (**5.3.2**) use **4.3.3**.

4.3.1 *Semi-micro steam distillation apparatus*, for example Hoskins's apparatus (80 mL)[1)] or Markham's apparatus (see BS 1428-B2).

4.3.2 *Funnel*, with long stem to reach below the ground joint of the apparatus described in **4.3.1**.

4.3.3 *Stillhead*, with tap funnel and splash bulb, in a rubber bung or with a ground joint to fit the Kjeldahl flasks (**4.2**), connected to a vertical condenser.

4.4 *Burette*, 10 mL, graduated in 0.02 mL.

5 Procedure

5.1 Test portion

Determine the non-ionic matter in accordance with BS 3762-3.7. Dissolve the weighed residue in the ethanol (**3.1**), transfer to the 100 mL one-mark volumetric flask (**4.1**) and dilute to the mark.

Pipette an aliquot portion (V mL) containing between 0.10 g and 0.18 g of non-ionic residue into the Kjeldahl flask (**4.2**). Alternatively, if the non-ionic residue is less than 0.2 g, take V = 50 mL. If V exceeds 25 mL pipette in two portions, evaporating the solvent after the first.

5.2 Digestion

Evaporate off the solvent. Add 2 g of the sodium sulphate (**3.3**) and 0.05 g of the selenium (**3.2**). Add 4.0 ± 0.2 mL of the sulphuric acid (**3.4**). Place the same quantities of reagents in a second Kjeldahl flask and proceed simultaneously with this blank determination.

Heat the contents of the flask to boiling. Boil gently until the carbonaceous matter is "oxidized" (about 20 min to 30 min), then boil for a further 20 min, gradually increasing the heat, so that the acid refluxes down the walls of the flask. Allow to cool.

NOTE The term "oxidized" has been used above in the absence of a more appropriate description.

To the cool Kjeldahl flask add 10 mL of water, using it to rinse the walls of the flask. Swirl to dissolve the sodium sulphate.

5.3 Distillation

Proceed in accordance with either **5.3.1** or **5.3.2**.

[1)] Hoskins, J. L. *Analyst*, 1944, 69,271.

5.3.1 *Steam distillation.* Pass steam through the semi-micro steam distillation apparatus (**4.3.1**), with water flowing through the condenser, for at least 20 min. Ensure that at least 3 mL/min of condensate can be produced.

Empty the inner vessel of the distillation apparatus by removing the source of heat from the steam generator, then drain, and close the outlet.

Unstopper the distillation apparatus, insert the funnel (**4.3.2**) into the inner chamber and add 20 ± 2 mL of the sodium hydroxide solution (**3.6**). Remove the funnel, taking care that no drop of alkali falls into the cup of the apparatus, and replace the stopper. Pass steam through the apparatus for 5 min to 10 min at a rate to produce about 3 mL of condensate per min.

Place 10 mL of the orthoboric acid solution (**3.5**) in a 100 mL conical flask, add 0.2 mL of the mixed indicator solution (**3.8**) and place the flask beneath the condenser. Raise the flask and add the minimum amount of water to ensure that the condenser tip is just covered.

Pour the contents of the Kjeldahl flask into the cup of the apparatus. Without interrupting the steam supply, carefully raise the stopper and allow the solution to flow into the inner chamber at such a rate that the orthoboric acid does not rise more than 20 mm to 30 mm into the condenser. Leave a few drops of liquid in the cup to maintain a seal. Rinse the Kjeldahl flask with 5 mL of water, transfer to the cup, and run into the inner chamber as before. Repeat the rinsing.

Allow distillation to continue for 5 min to 7 min, then lower the receiver and distil for a further 2 min to wash the inner surface of the condenser. Rinse the tip of the condenser with a little water. Continue in accordance with **5.4**.

For an immediate subsequent determination or for the blank determination, begin the distillation stage at paragraph 2 of this clause.

5.3.2 *Direct distillation.* Connect the Kjeldahl flask to the stillhead and condenser (**4.3.3**). Place 10 mL of the orthoboric acid solution (**3.5**) into a 100 mL conical flask, add 0.2 mL of the mixed indicator solution (**3.8**) and place the flask beneath the condenser. Raise the flask and add the minimum amount of water to ensure that the condenser tip is just covered.

Add 20 ± 2 mL of the sodium hydroxide solution (**3.6**) through the tap funnel into the flask, and rinse the funnel with 5 mL of water, maintaining a seal.

Heat the contents of the distillation flask to the boil, and boil until between one-half and two-thirds of the water present has distilled into the receiver. Lower the receiver during the distillation of the last few millimetres, then wash the tip of the condenser with water, collecting the washings in the receiver. Continue in accordance with **5.4**.

5.4 Titration

Titrate the contents of the receiver with the hydrochloric acid solution (**3.7**), using the burette (**4.4**).

6 Expression of results

The nitrogen content, N, of the non-ionic residue, expressed as a percentage by mass, is given by the following expression:

$$\frac{(V_1 - V_0) \times 0.1}{1000} \times \frac{100}{V} \times 14 \times \frac{100}{m}$$

$$= \frac{(V_1 - V_0) \times 14}{m \times V}$$

where

V_1 is the volume of the hydrochloric acid solution used to titrate the sample (in mL);

V_0 is the volume of the hydrochloric acid solution used to titrate the blank (in mL);

m is the mass of non-ionic residue (in g) (see BS 3762-3.7);

V is the volume of non-ionic solution taken (in mL).

The alkanolamides content of the sample, expressed as a percentage by mass, is given by the following expression:

$$\frac{N \times M \times P}{14 \times 100}$$

where

M is the assumed relative molecular mass of the alkanolamide, e.g. 243 if the result is expressed as dodecanoic ethanolamide;

P is the percentage non-ionic matter in the sample (see BS 3762-3.7).

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 3762-3.8:1986;

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

c) a complete identification of the sample.

Publications referred to

BS 1428, *Microchemical apparatus.*
BS 1428-B2, *Ammonia distillation apparatus (Markham).*
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 3978, *Water for laboratory use.*
Analyst, 1944, 69,271.

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