BRITISH STANDARD

**BS 3762-3.14:
1985**

Analysis of formulated detergents —

Part 3: Quantitative test methods —

Section 3.14 Method for determination of carbonate 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
January 2011

UDC 661.185:543.272:546.264

Foreword

This Section of BS 3762 has been prepared under the direction of the Chemicals Standards Committee and supersedes method D3 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 3762-3.14.

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.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 4, 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 Chemical Standards Committee, was published under the authority of the Board of BSI and comes into effect on
31 January 1985

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 84/50244 DC

ISBN 0 580 14129 2

Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

blank

1 Scope

This Section of BS 3762 describes a method of analysis for the determination of carbonate content of formulated detergents. The carbonate content may be expressed as carbon dioxide or as sodium carbonate.

NOTE The title of the publication referred to in this Section is given on the inside back cover.

2 Principle

Carbon dioxide is liberated from carbonate and bicarbonate present in the sample on the addition of acid. The carbon dioxide evolved is passed through solutions of chromic-sulphuric acid, to free it from sulphur dioxide, and a desiccant to free it from water. It is then absorbed in a weighed bulb containing an absorbent for carbon dioxide.
The bulb is then reweighed.

Chlorine bleaches, if present, are first destroyed by the addition of hydrogen peroxide.

3 Reagents

The reagents shall be of a recognized analytical reagent quality. Water complying with the requirements of BS 3978, from which carbon dioxide has been removed, for example by boiling, shall be used throughout.

3.1 *Hydrogen peroxide,* 6 % solution approximately.

3.2 *Absorbent* for carbon dioxide, granules, 12 mesh to 20 mesh.

NOTE "Carbosorb AB" is suitable. A self-indicating grade is preferred.

3.3 *Desiccant.* Calcium sulphate, anhydrous, granular, 8 mesh to 20 mesh.

NOTE A self-indicating grade is preferred.

3.4 *Antifoam agent.* Butanol or a suitable agent of the silicone type.

3.5 *Chromic-sulphuric acid solution.* Dissolve approximately 10 g of powdered potassium dichromate in 50 mL of water and add carefully approximately 950 mL of concentrated sulphuric acid (ρ = 1.84 g/mL).

3.6 *Hydrochloric acid,* c(HCl) = 5 mol/L approximately.

3.7 *Methyl orange indicator solution*

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

4 Apparatus

Ordinary laboratory apparatus and the following are required.

4.1 *Absorption train,* filled with reagents and assembled as illustrated in Figure 1, and comprising the following:

- a) *Guard tube,* straight type (A).
- b) *Dropping funnel,* of volume approximately 25 mL and with a long stem (B).
- c) *Flask,* flat-bottom, 250 mL or 500 mL (C).
- d) *Condenser* of short length, water-cooled (D).
- e) *Gas washing bottles,* Drechsel type (E), (F), (G).

  NOTE 1 Bottles of such a construction as to have integral back-suction traps are to be preferred (see note 2).
- f) *Absorption tube,* Fleming type (H). Alternatively a U-tube with two stop-cocks may be used.
- g) *Trap,* between the source of controlled vacuum and the absorption train.

NOTE 2 The volume of air space within the train should be kept to the minimum. Apparatus with ground-glass joints is preferred, and the joints should be well greased with a heavy stopcock grease. Other connections should be glass-to-glass, held in place with good-quality flexible plastics tubing; castor oil is a good lubricant and seal for such connections.
If the gas-washing bottles do not have integral back-suction traps, additional empty gas-washing bottles should be inserted between the condenser and vessel E and between the absorption tube H and vessel G, to act as safety traps.

5 Procedure

5.1 Test portion

Weigh, to the nearest 0.01 g, an appropriate amount of well mixed sample so as to contain 100 mg to 200 mg of carbon dioxide.

NOTE If the sample is a physical mixture of detergent and adjuncts and is difficult to sample accurately, it may be desirable to weigh a larger sample, dissolve in water, dilute to a specified volume and use a portion containing 100 mg to 200 mg of carbon dioxide.

5.2 Determination

Transfer the test portion to flask C and add 50 mL of water.

NOTE A lesser amount of water may be added if a liquid test portion is used.

Add a boiling aid and 10 mL of butanol or a small amount of other anti-foaming agent. If chlorine bleaches are present, add slowly sufficient hydrogen peroxide to destroy the bleach, i.e. until further additions produce no effervescence.

Insert the flask in the apparatus and draw a current of air through the train for a few minutes to sweep out all carbon dioxide; regulate the suction so that only two bubbles to four bubbles per second pass through the apparatus. With suction still on, close the tap of funnel B and check that there are no leaks; then discontinue the suction.

Close the taps of absorption tube H and remove it from the train. Temporarily block the tubing of the train to prevent ingress of moisture or carbon dioxide. Momentarily open and shut the inlet of H, then weigh the vessel. Re-insert the vessel into the train and open the taps. Fill funnel B with hydrochloric acid solution, add a few drops of methyl orange indicator and re-insert tube A.

Add the acid slowly to the test portion and then allow air to bubble through the train at two bubbles to four bubbles per second. Slowly heat the solution in the flask to boiling and continue boiling for 3 min to 4 min. The solution should now be red, indicating an excess of acid. If it is not, allow the flask to cool slightly, close the tap of funnel B and refill with the acid; re-insert tube A. Add the acid slowly and continue as previously.

After the boiling is discontinued, continue the passage of air at two bubbles to four bubbles per second through the system for at least 15 min. Close the taps of tube H and re-weigh it as before.

NOTE 1 It is recommended that the apparatus and technique should be checked occasionally with a sample of known carbonate content, e.g. anhydrous sodium carbonate which has been dried for 2 h at 105 °C.

NOTE 2 The carbon dioxide absorbing granules and desiccant should be changed when they are about half exhausted. The chromic-sulphuric acid should be replaced when a greenish colour develops.

6 Expression of results

The carbonate content, expressed as a percentage by mass of carbon dioxide (CO_2), is given by the expression

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

The carbonate content, expressed as a percentage by mass of sodium carbonate (Na_2CO_3), is given by the expression

$$\frac{m_1}{m_2} \times 240.8$$

where

m_1 is the increase in mass of absorption tube H (in g);

m_2 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.

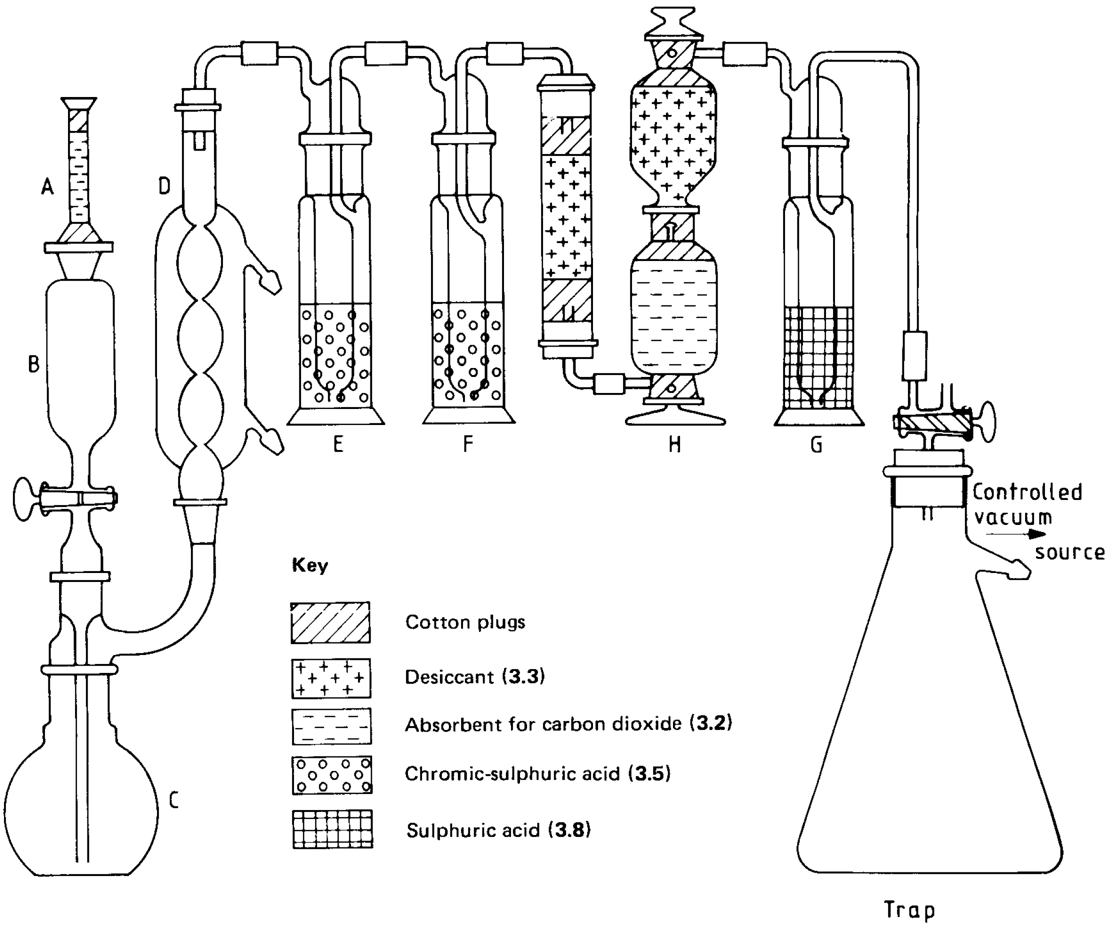

Figure 1 — Absorption train

blank

Publication referred to

BS 3978, *Water for laboratory use.*

BSI — British Standards Institution

BSI is the independent national body responsible for preparing British Standards. It presents the UK view on standards in Europe and at the international level. It is incorporated by Royal Charter.

Revisions

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions.

It is the constant aim of BSI to improve the quality of our products and services. We would be grateful if anyone finding an inaccuracy or ambiguity while using this British Standard would inform the Secretary of the technical committee responsible, the identity of which can be found on the inside front cover.
Tel: 020 8996 9000. Fax: 020 8996 7400.

BSI offers members an individual updating service called PLUS which ensures that subscribers automatically receive the latest editions of standards.

Buying standards

Orders for all BSI, international and foreign standards publications should be addressed to Customer Services. Tel: 020 8996 9001. Fax: 020 8996 7001.

In response to orders for international standards, it is BSI policy to supply the BSI implementation of those that have been published as British Standards, unless otherwise requested.

Information on standards

BSI provides a wide range of information on national, European and international standards through its Library and its Technical Help to Exporters Service. Various BSI electronic information services are also available which give details on all its products and services. Contact the Information Centre.
Tel: 020 8996 7111. Fax: 020 8996 7048.

Subscribing members of BSI are kept up to date with standards developments and receive substantial discounts on the purchase price of standards. For details of these and other benefits contact Membership Administration.
Tel: 020 8996 7002. Fax: 020 8996 7001.

Copyright

BSI
389 Chiswick High Road
London
W4 4AL