

Analysis of formulated detergents —

Part 3: Quantitative test methods —

Section 3.15 Method for estimation of carboxymethylcellulose 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.

UDC 661.185:543.42:547.458.8

Confirmed January 2011

Foreword

This Section of BS 3762 has been prepared under the direction of the Chemicals Standards Committee and supersedes method D4 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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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

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 31 January 1985

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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:

Committee reference CIC/34

Draft for comment 84/50245 DC

ISBN 0 580 14121 7

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1 Scope

This Section of BS 3762 describes a method of analysis for the estimation of carboxymethylcellulose content of formulated detergents.

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

2 Principle

Hydrolysis and dehydration of the carboxymethylcellulose to furfural derivatives, which produce a green colour with a solution of anthrone in 60 % sulphuric acid. The method is not specific for carboxymethylcellulose as most cellulose derivatives, and also other carbohydrates such as sucrose, react similarly. The intensity of the colour decreases with the increase in the degree of substitution of the carboxymethylcellulose and it is essential to prepare a standardization graph from similar material to that in the sample, or to express the result in terms of an arbitrary standard.

NOTE The method is based on that reported by Black, H.C. *Analytical Chemistry*, 1951, **23**, 1792-5.

3 Reagents

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

3.1 Kieselguhr

3.2 Sulphuric acid, 60 % (V/V) solution. Add 60 mL of the concentrated sulphuric acid ($\rho = 1.84 \text{ g/mL}$) slowly to 40 mL of water, cooling well. Dilute to 100 mL with water when cold.

3.3 Anthrone solution. Dissolve 0.2 g of anthrone ($\text{C}_6\text{H}_4\text{COC}_6\text{H}_4\text{CH}_2$) in about 50 mL of concentrated sulphuric acid ($\rho = 1.84 \text{ g/mL}$), which has been added to 5 mL of water. Cool and dilute to 200 mL with the concentrated sulphuric acid. Allow the solution to stand for 4 h before use. Do not use a solution that is more than 24 h old.

4 Apparatus

Ordinary laboratory apparatus and the following are required.

4.1 One-mark volumetric flasks of capacity 50, 100 and 200 mL, complying with the requirements of BS 1792.

4.2 Filter, sintered or fritted glass or silica, porosity grade no. 4, complying with the requirements of BS 1752.

4.3 Spectrometer (or photoelectric absorptiometer fitted with filters providing maximum transmission at a wavelength of about 625 nm) and a cell of optical path length 10 mm.

5 Procedure

5.1 Test portion

Weigh 1.00 g of the well mixed test sample.

5.2 Determination

Dissolve the test portion in the sulphuric acid solution (3.2), and, using this acid, dilute to 100 mL in a one-mark volumetric flask. If per-salts are present, heat the test portion for 2 h in an oven at 150 °C and then allow it to cool, before dissolving in the acid.

Filter the solution through the filter (4.2), using a little kieselguhr (3.1) to aid filtration. Transfer 5 mL of the filtered solution to a 50 mL one-mark volumetric flask.

Add 80 mL of the anthrone solution (3.3) in a slow continuous stream to 50 mL water while stirring. Add 30 mL of the resulting warm solution to the 50 mL flask containing the sample solution and to a second flask for a blank. Heat the flasks in a boiling water bath for 15 min. Cool and dilute each to the mark with the sulphuric acid (3.2). Measure the absorbance of the sample solution in a cell of optical path length 10 mm at 625 nm against the blank.

Prepare a standardizing graph from the figures obtained by treating known amounts of carboxymethylcellulose (see clause 2) by the above procedure, to cover a suitable range.

6 Expression of results

Record the percentage of carboxymethylcellulose in the sample by reference to the graph.

7 Test report

The test report shall include the following information:

- a reference to this British Standard, i.e. BS 3762-3.15;
- the results and the method of expression used;
- the test conditions.

Publications referred to

BS 1752, *Laboratory sintered or fritted filters.*

BS 1792, *One-mark volumetric flasks.*

BS 3978, *Water for laboratory use.*

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