BRITISH STANDARD

BS 1715-2.9: 1990

Analysis of soaps —

Part 2: Quantitative test methods —

Section 2.9 Method for determination of EDTA content

Confirmed January 2011

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Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Chemicals Standards Policy Committee (CIC/-) to Technical Committee CIC/34, upon which the following bodies were represented:

Chemical Industries Association
Chemical Industries Association (GOSIP)
Consumer Policy Committee of BSI
Department of Trade and Industry (Laboratory of the Government Chemist)
Ministry of Defence
Royal Society of Chemistry
Soap and Detergent Industry Association
Society of Dyers and Colourists

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Foreword

This Section of BS 1715 has been prepared under the direction of the Chemicals Standards Policy Committee.

During the preparation of this Section of BS 1715, account was taken of ISO 4325:1977 "*Soaps and detergents — Determination of EDTA content (sequestering agent) — Titrimetric method*", published by the International Organization for Standardization (ISO). The principal differences between this method and that given in ISO 4325 are that they use different indicator systems and that this Section of BS 1715 includes a procedure for precipitation and removal of the soap fatty matter.

This Section describes a method of test only, and should not be used or quoted as a specification defining limits of purity. Reference to this Section should indicate that the method of test used is in accordance with BS 1715-2.9.

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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.

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1 Scope

This Section of BS 1715 describes a titrimetric method for the determination of the content of ethylenediaminetetra-acetic acid (EDTA) and its salts in hard soap, soft soap, toilet soap and soap flakes. It is applicable to soaps with EDTA contents of 0.01 % to 0.1 %.

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

2 Principle

The fatty matter is precipitated and removed from an aqueous solution of the soap. This solution is adjusted to pH 4.6 and then titrated with standard copper(II) sulphate solution using 1-(2-pyridylazo)-2-naphthol (PAN) as indicator.

3 Reagents

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

3.1 *Hydrochloric acid solution*, c(HCl) = 5 mol/L.

3.2 *Sodium hydroxide solution*, approximately 40 g/L.

3.3 *Copper(II) sulphate standard volumetric solution*, $c(CuSO_4)$ = 0.100 mol/L.

3.4 *PAN indicator*, 1 g/L solution of 1-(2-pyridylazo)-2-naphthol in ethanol.

NOTE For the purposes of **3.4**, the ethanol may be replaced by industrial methylated spirits complying with BS 3591. 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 accepted alternative.

4 Apparatus

Ordinary laboratory apparatus and the following are required.

4.1 *pH meter with electrodes and stirrer*

4.2 *Steam bath, or other heating equipment*, capable of being maintained at 100 ± 5 °C.

4.3 *Burette*, 10 mL capacity, with graduation intervals of 0.02 mL, and complying with class A of BS 846.

5 Sampling

The laboratory sample shall have been taken and prepared in accordance with BS 1715-1.

6 Procedure

6.1 Test portion

Weigh, to the nearest 0.01 g, into a squat form beaker, a quantity of the laboratory sample sufficient to give an expected titration of between 1 mL and 5 mL [e.g. at an expected EDTA content of 0.02 % (m/m), weigh 30 g of the sample].

6.2 Removal of the fatty matter

Add approximately 200 mL of hot water to the test portion, place the beaker on the steam bath or other heater maintained at 100 ± 5 °C (**4.2**) and stir to dissolve. While stirring, add a small excess of the hydrochloric acid solution (**3.1**) to precipitate the soap fatty matter.

NOTE For typical samples, approximately 6 mL of the hydrochloric acid solution (**3.1**) is required for each 10 g of laboratory sample.

Continue heating until the fatty matter has formed a clear layer at the top, cool and filter into a second beaker through a fast or medium/fast filter paper, previously wetted with water, and wash with hot water. Discard the fatty matter.

6.3 Determination

While stirring, introduce the electrodes connected to the pH meter (**4.1**), which has been standardized, and carefully add the sodium hydroxide solution (**3.2**) until the pH is 4.6 ± 0.5. Raise the electrodes and rinse them into the beaker.

Add 0.4 mL of the PAN indicator (**3.4**) and, while stirring, and using the burette (**4.3**), titrate with the copper(II) sulphate solution (**3.3**) until the colour changes from yellow to wine red.

NOTE The wine red colour should persist for at least 1 min.

6.4 Blank determination

Carry out a blank determination by titrating 200 mL of water adjusted to pH 4.6 ± 0.5 until the colour of the blank determination and that of the sample determination are the same.

7 Expression of results

Subtract the volume of the copper(II) sulphate solution used for the blank determination from the volume used for the sample determination, to produce the corrected titration volume V.

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

$$\frac{V \times 0.01}{1000} \times 292 \times \frac{100}{m}$$

where

V is the corrected titration volume of the copper(II) sulphate solution used (in mL);

m is the mass of the test portion (in g);

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292 is the relative molecular mass of EDTA (free acid).

8 Precision

No precision data are available.

9 Test report

The test report shall include the following information:

a) a reference to this British Standard, i.e. BS 1715-2.9:1990;

b) the results expressed in accordance with clause **7**;

c) a complete identification of the sample.

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Publication(s) referred to

BS 846, *Specification for burettes.*

BS 1715, *Analysis of soaps.*

BS 1715-1, *General introduction, sampling, and test for presence of synthetic anionic-active surface active agents.*

BS 3591, *Specification for industrial methylated spirits.*

BS 3978, *Specification for water for laboratory use.*

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