BRITISH STANDARD

BS 1715-2.2:
1989
ISO 684:1974

Analysis of soaps —

Part 2: Quantitative test methods —

Section 2.2 Method for determination of total free alkali content

[ISO title: Analysis of soaps — Determination of total free alkali]

UDC 661.18:661.3:543

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
Consumer Policy Committee of BSI
Department of the Environment
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

This British Standard, having been prepared under the direction of the Chemicals Standards Policy Committee, was published under the authority of the Board of BSI and comes into effect on 31 July 1989

The following BSI references relate to the work on this standard:
Committee reference CIC/34
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Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

National foreword

This Section of BS 1715 has been prepared under the direction of the Chemicals Standards Policy Committee. It is identical with ISO 684:1974 "*Analysis of soaps — Determination of total free alkali*" published by the International Organization for Standardization (ISO).

This method supersedes the method for determination of total free alkali given in BS 1715:1963, which is deleted by amendment.

Terminology and conventions. The text of the International Standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

The symbol "ml" has been used to denote millilitre. In British Standards it is current practice to use the symbol "mL".

Wherever the words "International Standard" appear, referring to this standard, they should be read as "Section of BS 1715".

Cross-reference

International Standard	Corresponding British Standard
ISO 685:1975	BS 1715 *Analysis of soaps* Section 2.1:1989 *Method for determination of total alkali content and total fatty matter content* (Identical)

The International Standard for sampling of soaps (see clause **2** and clause **7**) is not yet published. Relevant information is included in BS 1715 "*Analysis of soaps*", Part 1:1989 "*General introduction, sampling, and test for presence of synthetic anionic-active surface active agents*".

Additional information. With reference to clause **5**, water complying with grade 3 of BS 3978 "*Specification for water for laboratory use*" is suitable.

With reference to **5.2**, the sulphuric acid standard volumetric solution, expressed as an amount-of-substance concentration, is $c(\frac{1}{2}H_2SO_4) = 1$ mol/L. With reference to **5.3**, the potassium hydroxide standard volumetric solution, expressed as an amount-of-substance concentration, is c(KOH) = 0.1 mol/L. In the definitions of T_0 and T_1 in **9.1**, "normality" should be read as "amount-of-substance concentration (mol/L)". In the penultimate paragraph of **9.1**, "milliequivalents" should be read as "millimoles".

For the purposes of **5.1** the ethanol may be replaced by industrial methylated spirits complying with BS 3591 "*Specification for industrial methylated spirits*" of appropriate strength. 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.

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 1715-2.2.

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 to iv, 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 and field of application

This International Standard specifies a method for the determination of the total free alkali content of commercial soaps, excluding compounded products[1].

The method is not applicable if the soap contains additives (alkali silicates, etc.) which can be decomposed by sulphuric acid by the procedure specified. The method is also not applicable to coloured soaps if the colour interferes with the phenolphthalein end point.

2 References

ISO 685, *Analysis of soaps — Determination of total alkali*[2].

ISO . . ., *Soaps — Sampling*[3].

3 Definition

For the purpose of this International Standard, the following definition applies:

3.1
total free alkali

the sum of the free caustic alkali and the free carbonate alkali contents

the results are generally expressed as a percentage by mass as either sodium hydroxide (NaOH) for sodium soaps or potassium hydroxide (KOH) for potassium soaps

they may also be expressed in milliequivalents per gram

4 Principle

Dissolution of the soap in an ethanolic solution, neutralization of the free alkali with a known excess of sulphuric acid solution and back titration of the excess acid with ethanolic potassium hydroxide solution.

5 Reagents

During the analysis, use only reagents of recognized analytical reagent grade and only distilled water or water of equivalent purity.

5.1 *Ethanol,* 95 % (*V*/*V*) solution, free from carbon dioxide.

Reflux this solution for 5 min, cool to ambient temperature and neutralize with the ethanolic potassium hydroxide solution (**5.3**) in the presence of 4 drops of the phenolphthalein solution (**5.4**) per 200 ml of ethanol.

5.2 *Sulphuric acid,* approximately N standard volumetric solution.

5.3 *Potassium hydroxide,* approximately 0,1 N ethanolic standard volumetric solution.

5.4 *Phenolphthalein,* solution of 1 g per 100 ml in 95 % (*V*/*V*) ethanol.

6 Apparatus

Ordinary laboratory apparatus and

6.1 *Conical flask,* capacity 250 ml, with a conical ground glass joint.

6.2 *Reflux condenser,* water-cooled, with a conical ground glass joint at the bottom.

7 Sampling

Laboratory samples shall be prepared and stored according to the procedures specified in ISO...

8 Procedure

8.1 Test portion

Weigh, to the nearest 0,001 g, about 5 g of the laboratory sample into the conical flask (**6.1**).

8.2 Determination

Add 100 ml of the ethanol solution (**5.1**) to the test portion (**8.1**).

Fit the conical flask (**6.1**) to the reflux condenser (**6.2**). Gently heat until the soap is completely dissolved, then add exactly 3,0 ml [4] of the sulphuric acid solution (**5.2**) and boil gently for at least 10 min. Allow to cool to ambient temperature.

Titrate with the ethanolic potassium hydroxide solution (**5.3**) in the presence of the indicator (**5.4**).

Carry out two determinations on the same sample.

9 Expression of results

9.1 Method of calculation and formulae

The total free alkali content, expressed as a percentage by mass of sodium hydroxide (NaOH) in the case of sodium soaps, is given by the formula:

$$0{,}040 \times \frac{V_0 T_0 - V_1 T_1}{m} \times 100$$

[1] See also ISO 685.
[2] At present at the stage of draft. (Revision of ISO/R 685.)
[3] In preparation.
[4] This volume may be increased for certain soaps with high total free alkali contents.

The total free alkali content, expressed as a percentage by mass of potassium hydroxide (KOH) in the case of potassium soaps, is given by the formula:

$$0{,}056 \times \frac{V_0 T_0 - V_1 T_1}{m} \times 100$$

where

m is the mass, in grams, of the test portion (**8.1**);

V_0 is the volume, in millilitres, of the sulphuric acid solution (**5.2**) used in the determination;

V_1 is the volume, in millilitres, of the potassium hydroxide solution (**5.3**) used in the titration;

T_0 is the exact normality of the sulphuric acid solution (**5.2**);

T_1 is the exact normality of the potassium hydroxide solution (**5.3**).

The total free alkali may also be expressed in milliequivalents per gram by the formula:

$$\frac{V_0 T_0 - V_1 T_1}{m}$$

Take as the result the arithmetic mean of the duplicate determinations.

9.2 Reproducibility

The results obtained on the same sample in two different laboratories, expressed either as sodium hydroxide (NaOH) or as potassium hydroxide (KOH), shall not differ by more than 0,05 %.

10 Test report

The test report shall include the following particulars:

a) all information necessary for the complete identification of the sample;

b) the reference to method used, reference being made to this International Standard;

c) the results obtained and the method of expression used;

d) the test conditions;

e) any operational details not specified in this International Standard, or regarded as optional, as well as all incidents likely to have affected the results.

Publications referred to

See national foreword.

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