BRITISH STANDARD

BS 1715-2.1:
1989
ISO 685:1975

Analysis of soaps —

Part 2: Quantitative test methods —

Section 2.1 Method for determination of total alkali content and total fatty matter content

[ISO title: Analysis of soaps — Determination of total alkali content and total fatty matter content]

UDC 661.18:661.187.2:543

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
Draft for comment 88/53280 DC

ISBN 0 580 17287 2

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 685:1975 "*Analysis of soaps — Determination of total alkali content and total fatty matter content*" published by the International Organization for Standardization (ISO).

This method supersedes the methods for determination of total fatty matter and determination of total alkali given in BS 1715:1963, which are 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 "l" has been used to denote litre (and in its submultiples). In British Standards it is current practice to use the symbol "L".

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 684:1974	BS 1715 *Analysis of soaps* Section 2.2:1989 *Method for determination of total free alkali content* (Identical)

The International Standard for sampling of soaps (see clause **2** and clause **7**) is not yet published. Relevant information on sampling 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 **5.4**, the sulphuric acid or hydrochloric acid standard volumetric solution, expressed as an amount-of substance concentration, is $c(\frac{1}{2}H_2SO_4) = 1$ mol/L or $c(HCl) = 1$ mol/L. With reference to **5.5** and **5.6**, the sodium hydroxide and potassium hydroxide standard volumetric solutions, expressed as amount-of-substance concentrations, are $c(NaOH) = 1$ mol/L and $c(KOH) = 1$ mol/L, respectively. In the definitions of T_0 and T_1 in **9.1.1**, and T in **9.2.1**, "normality" should be read as "amount-of-substance concentration (mol/L)".

For the purposes of **5.3** 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 1715-2.1.

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 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.

blank

1 Scope and field of application

This International Standard specifies a method for the simultaneous determination of the total alkali[1)] content and the total fatty matter content of soaps, excluding compounded products.

This method for the determination of total alkali is not applicable to coloured soaps if the colour interferes with the methyl orange end-point.

2 References

ISO 684, *Analysis of soaps — Determination of total free alkali.*

ISO ..., *Soaps — Sampling*[2)].

3 Definitions

For the purposes of this International Standard, the following definitions apply:

total alkali

the sum of the alkali bases combined as soap with fatty and rosin acids, as well as those corresponding to free alkali metal hydroxides or carbonates and to any silicates present which will be titrated under the test conditions

the results are expressed as a percentage by mass of either sodium hydroxide (NaOH) or of potassium hydroxide (KOH), according to whether sodium or potassium soaps are concerned

total fatty matter

the water-insoluble fatty material obtained by decomposing the soap with a mineral acid under the conditions specified. This term includes unsaponifiable matter, glycerides and any rosin acids contained in the soap, in addition to the fatty acids

4 Principle

Decomposition of the soap by a known volume of standard volumetric mineral acid solution, extraction and separation of the liberated fatty matter with light petroleum and determination of the total alkali content by titration of the excess of acid contained in the aqueous phase with a standard volumetric sodium hydroxide solution. After evaporation of the light petroleum from the extract, dissolution of the residue in ethanol and neutralization of the fatty acids with a standard volumetric potassium hydroxide solution. Evaporation of the ethanol and weighing of the soap formed to determine the total fatty matter content.

5 Reagents

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

5.1 *Acetone*

5.2 *Light petroleum,* boiling range between 40 and 60 °C.

5.3 *Ethanol,* 95 % (*V*/*V*) solution, neutralized to the phenolphthalein solution (**5.8**).

5.4 *Sulphuric acid or hydrochloric acid,* approximately 1 N standard volumetric solution

5.5 *Sodium hydroxide,* approximately 1 N standard volumetric solution, standardized using the methyl orange solution (**5.7**) as indicator.

5.6 *Potassium hydroxide,* approximately 1 N standard volumetric solution in ethanol (**5.3**).

5.7 *Methyl orange,* 2 g/l solution.

5.8 *Phenolphthalein,* 10 g/l solution in ethanol (**5.3**).

6 Apparatus

Ordinary laboratory apparatus and

6.1 *Beaker,* capacity 250 ml, squat form, complying with ISO 3819.

6.2 *Separating funnels,* capacity 500 ml, or

6.3 *Extraction cylinder,* capacity 250 ml, diameter 39 mm and height 355 mm, fitted with a ground glass stopper.

6.4 *Water bath*

6.5 *Oven,* capable of being controlled at 103 ± 2 °C.

7 Sampling

The laboratory sample of soap shall be prepared and stored in accordance with the instructions given in ISO...

8 Procedure

Carry out two determinations on the same sample.

8.1 Test portion

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

8.2 Determination

Dissolve the test portion (**8.1**) in about 100 ml of hot water.

Pour the solution into one of the separating funnels (**6.2**) or into the extraction cylinder (**6.3**) and wash the beaker with small quantities of water, adding the washings to the separating funnel or to the extraction cylinder.

[1)] See also ISO 684.

[2)] In preparation.

Add a few drops of the methyl orange solution (**5.7**) and then, from a burette, add, while vigorously shaking the separating funnel or the extraction cylinder, an accurately measured known volume of the sulphuric acid or hydrochloric acid solution (**5.4**) until there is an excess of about 5 ml. Cool the contents of the separating funnel or of the extraction cylinder to about 25 °C and add 100 ml of the light petroleum (**5.2**). Insert the stopper and gently invert the separating funnel or the extraction cylinder, whilst maintaining a hold on the stopper. Open the stopcock of the separating funnel or the stopper of the extraction cylinder gradually to release any pressure, then close, gently shake and again release the pressure. Repeat the shaking until the aqueous layer has become clear, and then allow to stand.

a) *In the case of use of separating funnels*

Run off the aqueous layer into a second separating funnel (**6.2**) and extract with 50 ml of the light petroleum (**5.2**). Repeat the process, collect the aqueous layer in a conical flask and combine the three light petroleum extracts in the first separating funnel.

b) *In the case of use of an extraction cylinder*

Using a siphon, draw off the light petroleum layer as completely as possible into a separating funnel (**6.2**).

Repeat the extraction twice with 50 ml of the light petroleum (**5.2**), combine the three light petroleum extracts in the separating funnel, transfer the aqueous layer as completely as possible to a conical flask and wash the extraction cylinder with small quantities of water, adding the washings to the conical flask.

Wash the light petroleum extract by shaking with water (about 25 ml) until the washings are neutral to the methyl orange solution (**5.7**). Usually three washings are sufficient.

NOTE Allow each wash to stand for at least 5 min or such a time as is required to give a clear line of demarcation between the layers, before running off the aqueous layer.

After the final washing has been run off, impart a swirling motion to the contents of the separating funnel by rotating it sharply, but without inverting it, to remove any water droplets adhering to the sides.

Allow to stand for at least 5 min and run off any separated water.

Collect the washings of the light petroleum extract quantitatively in the conical flask already containing the aqueous layer.

8.2.1 *Determination of total alkali content*

Titrate the mixed acid aqueous layer and washings with the sodium hydroxide solution (**5.5**) using the methyl orange solution (**5.7**) as indicator.

8.2.2 *Determination of total fatty matter content*

Carefully transfer the washed light petroleum solution to a weighed, flat-bottomed flask, filtering if necessary through a dry filter paper. Wash the separating funnel with two or three small quantities of the light petroleum and filter the washings into the flask, taking precautions to prevent evaporation of the light petroleum during the filtration. Thoroughly wash the filter with the light petroleum, collecting the washings in the flask.

Evaporate off nearly all the light petroleum on the water bath (**6.4**), taking all necessary precautions, and under a slow stream of cold dry nitrogen or air.

Dissolve the residue in 20 ml of the ethanol (**5.3**), add a few drops of the phenolphthalein solution (**5.8**) and titrate with the ethanolic potassium hydroxide solution (**5.6**) to a faint permanent pink colour. Note the volume used.

Evaporate the ethanolic solution on the water bath (**6.4**). When the evaporation is near completion, rotate the flask in order to distribute the potassium soap in a thin layer on the sides and bottom of the vessel.

Carry out a preliminary drying of the potassium soap by adding acetone (**5.1**) and evaporating off the acetone on the water bath under a slow stream of cold dry nitrogen or air. Then heat to constant mass in the oven (**6.5**), controlled at 103 ± 2 °C, i.e. until the difference in mass after heating for an additional 15 min does not exceed 3 mg. Cool in a desiccator and weigh.

9 Expression of results

9.1 Total alkali content

9.1.1 *Method of calculation and formulae*

The total alkali content is given, as a percentage by mass, by the formulae:

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

expressed as sodium hydroxide (NaOH) for sodium soaps, and

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

expressed as potassium hydroxide (KOH) for potassium soaps, and

where

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

V_0 is the volume, in millilitres, of the standard volumetric acid solution (**5.4**) used;

V_1 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (**5.5**) used;

T_0 is the exact normality of the standard volumetric acid solution (**5.4**);

T_1 is the exact normality of the standard volumetric sodium hydroxide solution (**5.5**).

The total alkali content 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.

9.1.2 ***Reproducibility***

The difference between the results obtained on the same sample in two different laboratories should not differ by more than 0,2 % from the value of the percentage by mass of total alkali found, expressed either as sodium hydroxide or as potassium hydroxide.

9.2 Total fatty matter content

9.2.1 ***Method of calculation and formula***

The total fatty matter content is given, as a percentage by mass, by the formula:

$$[m_1 - (V \times T \times 0{,}038)] \times \frac{100}{m_0}$$

where

m_0 is the mass, in grams of the test portion (**8.1**);

m_1 is the mass, in grams, of the dried potassium soap;

V is the volume, in millilitres, of the standard volumetric ethanolic potassium hydroxide solution (**5.6**) used for the neutralization;

T is the exact normality of the standard volumetric ethanolic potassium hydroxide solution (**5.6**) used.

Take as the result the arithmetic mean of the duplicate determinations, rounding the result to the nearest 0,1 %.

9.2.2 ***Reproducibility***

The difference between the results obtained on the same sample in two different laboratories should not differ by more than 0,2 % from the value of the percentage by mass of total fatty matter found.

10 Test report

The test report shall include the following particulars:

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.

blank

Publication referred to

See national foreword.

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