BRITISH STANDARD

BS 1715-2.5: 1989
ISO 1067:1974

Analysis of soaps —

Part 2: Quantitative test methods —

Section 2.5 Method for determination of unsaponifiable, unsaponified, and unsaponified saponifiable matter contents

[ISO title: Analysis of soaps — Determination of unsaponifiable, unsaponified and unsaponified saponifiable matter]

UDC 661.18:543

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:
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Contents

		Page
Committees responsible		Inside front cover
National foreword		ii
1	Scope	1
2	Field of application	1
3	Principle	1
4	Reagents	1
5	Apparatus	1
6	Sampling	1
7	Procedure	1
8	Expression of results	2
9	Test report	2
Publications referred to		Inside back cover

National foreword

This Section of BS 1715 has been prepared under the direction of the Chemicals Standards Policy Committee. It is identical with ISO 1067:1974 "*Analysis of soaps — Determination of unsaponifiable, unsaponified and unsaponified saponifiable matter*" published by the International Organization for Standardization (ISO).

This method supersedes the methods for determination of unsaponified neutral fat and unsaponifiable matter, 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 "relative molar mass" appear, they should be read as "relative molecular mass".

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 1773:1976 (replacing ISO/R 1773)	BS 2734:1984 *Specification for boiling flasks (narrow-necked), conical, flat bottom and round bottom* (Identical)

With reference to **5.5**, a British Standard related to ISO/R 648 (now revised as ISO 648:1977) is BS 1583:1986 "*Specification for one-mark pipettes*".

The International Standard for sampling of soaps (see clause **6**) 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 **4**, water complying with grade 3 of BS 3978 "*Specification for water for laboratory use*" is suitable, and the reagents used should be of a recognized analytical grade, unless otherwise specified.

For the purposes of **4.1**, **4.4**, **4.5** and **4.6**, 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 (SI 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.

With reference to **4.4** and **4.5**, the potassium hydroxide standard volumetric solutions, expressed as amount-of-substance concentrations, are $c(\mathrm{KOH}) = 0.1$ mol/L and $c(\mathrm{KOH}) = 2$ mol/L respectively.

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 1715-2.5.

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

This International Standard specifies a method for the determination of the contents of unsaponifiable, unsaponified and unsaponified saponifiable matter in commercial soaps, excluding compound products.

2 Field of application

This method is applicable to the determination of the contents of products, other than free fatty acids, which are soluble in hexane or light petroleum (unsaponifiable + unsaponified matter), and which can be saponified (unsaponified saponifiable matter).

The method is not applicable to soaps enriched with sterols or long chain alcohols, nor to soaps containing perfume.

3 Principle

Extraction of matter soluble in hexane, and titration of the free fatty acids removed, with potassium hydroxide solution.

Saponification of products soluble in hexane neutralized in this way and extraction of the unsaponifiable matter by hexane.

4 Reagents

The water used shall be distilled water or water of equivalent purity. The reagents shall have the following characteristics:

4.1 *Ethanol*, free from carbon dioxide, neutralized hot with the ethanolic potassium hydroxide solution (**4.4**) using the phenolphthalein solution (**4.6**) as indicator.

4.2 *Sodium hydrogen carbonate*, 10 g/l solution.

4.3 *n-Hexane*, technical grade or, failing this, *light petroleum* distilling at between 40 and 60 °C, having a bromine number less than 1, and free from residue.

4.4 *Potassium hydroxide*, 0,1 N standard volumetric solution in ethanol.

4.5 *Potassium hydroxide*, 2 N standard volumetric solution in ethanol.

4.6 *Phenolphthalein*, 10 g/l solution in 95 % (*V*/*V*) ethanol.

5 Apparatus

Ordinary laboratory apparatus and, in particular:

5.1 *Beaker*, 250 ml.

5.2 *Separating funnels*, 50 ml and 250 ml.

5.3 *Round-bottomed flasks*, 100 ml and 250 ml, complying with ISO/R 1773.

5.4 *Microburette*, 2 ml.

5.5 *Pipette*, 10 ml, complying with ISO/R 648.

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

6 Sampling

The laboratory sample of soap shall be prepared and stored in accordance with the instructions given in ISO . . ., *Soaps — Sampling*.[1)]

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,001 g, about 5 g of the laboratory sample, the soap being finely grated, into the 250 ml beaker (**5.1**).

7.2 Determination

Add 50 ml of the neutralized ethanol (**4.1**) and 50 ml of the sodium hydrogen carbonate solution (**4.2**) to the test portion (**7.1**). Dissolve the soap by heating to not above 70 °C.

After the soap is completely dissolved, allow the solution to cool. Transfer the solution quantitatively to the 250 ml separating funnel (**5.2**), rinsing the beaker several times with a mixture of equal volumes of the neutralized ethanol (**4.1**) and the sodium hydrogen carbonate solution (**4.2**), and extract three times, stirring carefully, each time with 50 ml of the hexane or the light petroleum (**4.3**). Combine the extracts, filter them if necessary, and wash them until neutral to phenolphthalein, using for each wash 50 ml of a mixture of equal volumes of the neutralized ethanol (**4.1**) and water. Normally three washings are sufficient. Transfer the solution quantitatively to the 250 ml flask (**5.3**), previously dried in the oven (**5.6**) controlled at 103 ± 2 °C, allowed to cool in a desiccator and weighed to the nearest 0,2 mg.

Evaporate most of the solvent on a boiling water bath and remove the last traces with the aid of a gentle current of dry air directed into the flask while it is held obliquely, almost entirely immersed in the bath, and rotated.

[1)] In preparation.

Dry the flask and residue for 5 min in the oven (**5.6**) controlled at 103 ± 2 °C, allow it to cool in a desiccator and weigh to the nearest 0,2 mg. Repeat the operations of drying, cooling and weighing until the difference between two successive weighings does not exceed 2 mg. Let this mass be m_1.

Dissolve the residue in a few millilitres of the neutralized ethanol (**4.1**). Using the microburette (**5.4**), titrate the free acidity with the potassium hydroxide solution (**4.4**), using the phenolphthalein solution (**4.6**) as indicator, until the solution turns pink. Note the volume V of this solution used for the titration.

Add 10,0 ml of the potassium hydroxide solution (**4.5**), using the pipette (**5.5**). Bring the solution to boiling point and boil it under reflux for 30 min. Then add a volume of water equal to the volume of the solution and transfer the solution quantitatively to the 50 ml separating funnel (**5.2**), using a few millilitres of a mixture of equal volumes of the neutralized ethanol (**4.1**) and water to rinse the flask. Extract three times, each time with 10 ml of the hexane or the light petroleum (**4.3**). Combine the extracts and wash them until neutral to phenolphthalein, using for each wash 10 ml of a mixture of equal volumes of the neutralized ethanol (**4.1**) and water. Normally three washings are sufficient. Transfer the solution quantitatively into the 100 ml flask (**5.3**), previously dried in the oven (**5.6**) controlled at 103 ± 2 °C, allowed to cool in a desiccator and weighed to the nearest 0,2 mg.

Evaporate most of the solvent on a boiling water bath and remove the last traces with the aid of a gentle current of dry air directed into the flask while it is held obliquely, almost entirely immersed in the bath, and rotated.

Dry the flask and residue for 5 min in the oven (**5.6**) controlled at 103 ± 2 °C, allow it to cool in a desiccator and weigh to the nearest 0,2 mg. Repeat the operations of drying, cooling and weighing until the difference between two successive weighings does not exceed 2 mg. Let this mass be m_2.

8 Expression of results

The percentage, by mass, of unsaponified matter + unsaponifiable matter in the soap is equal to

$$\left(m_1 - \frac{V \times M}{10\,000}\right) \times \frac{100}{m_0}$$

The percentage, by mass, of unsaponifiable matter in the soap is equal to

$$m_2 \times \frac{100}{m_0}$$

The percentage, by mass, of unsaponified saponifiable matter in the soap is equal to

$$\left(m_1 - \frac{V \times M}{10\,000} - m_2\right) \times \frac{100}{m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the first extract;

m_2 is the mass, in grams, of the second extract;

M is the mean relative molar mass of the fatty acids of the soap (see note);

V is the volume, in millilitres, of the 0,1 N standard volumetric ethanolic potassium hydroxide solution (**4.4**) used to determine the acidity of the first extract.

NOTE The mean relative molar mass M of the fatty acids of the soap can be determined by titration of the fatty acids isolated after the saponification of a sample of the original soap, the elimination of unsaponifiable matter and the acidification of the soap solution.

9 Test report

The test report shall indicate the method used and the results obtained. It shall also mention all test conditions and procedural details not specified in this International Standard, or which are optional, and any factors which may have affected the results.

The test report shall give all information necessary for the complete identification of the sample.

Publications referred to

See national foreword.

BSI — British Standards Institution

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