BRITISH STANDARD

BS 1715-2.11: 1989
ISO 673:1981

Analysis of soaps —

Part 2: Quantitative test methods —

Section 2.11 Method for determination of ethanol-insoluble matter content

[ISO title: Soaps — Determination of content of ethanol-insoluble 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:
Committee reference CIC/34
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Amendments issued since publication

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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 673:1981 "*Soaps — Determination of content of ethanol-insoluble matter*" published by the International Organization for Standardization (ISO).

This method supersedes the method "Determination of matter insoluble in ethanol" 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".

Additional information. For the purposes of **4.1**, 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 (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 clause **6**, 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.*"

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 1715-2.11.

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.

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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 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.

1 Scope and field of application

This International Standard specifies a method for the determination of the content of ethanol-insoluble matter in commercial soaps, excluding compounded products.

2 Definition

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

ethanol-insoluble matter
the matter not dissolved by the procedure specified in this International Standard

NOTE 1 The ethanol-insoluble matter corresponds to the additives and foreign matter, of low solubility or insoluble in 95 % (*V*/*V*) ethanol, added to soaps, and also to substances in all soap formulations, such as alkali carbonates and chlorides, of low solubility in 95 % (*V*/*V*) ethanol.

NOTE 2 The foreign matter may be inorganic (carbonates, borates, perborates, chlorides, sulphates, silicates, phosphates, iron oxides, etc.) or organic (starches, dextrins, caseins, sugars, cellulose derivatives, alginates, etc.).

3 Principle

Dissolution of the soap in ethanol, filtration and weighing of the undissolved residue.

4 Reagent

4.1 *Ethanol*, 95 % (*V*/*V*).

5 Apparatus

Ordinary laboratory apparatus and

5.1 *Conical flasks*, of capacity 500 ml, having ground glass necks.

5.2 *Reflux condenser*, water-cooled, with a conical ground glass joint at the bottom to fit the conical flasks (**5.1**).

5.3 *Water bath*

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

5.5 *Analytical balance*, accurate to 0,001 g.

6 Sampling

Procedures for the preparation and storage of the laboratory sample will form the subject of a future International Standard.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,01 g, about 5 g of the laboratory sample into a conical flask (**5.1**).

7.2 Determination

Add 200 ml of the ethanol (**4.1**) to the test portion (**7.1**) in the conical flask and connect the reflux condenser (**5.2**).

Heat to gentle boiling, swirling in order to avoid, as far as possible, material adhering to the bottom of the flask.

Dry the filter paper to be used for the filtration of the insoluble matter in the oven (**5.4**), controlled at 103 ± 2 °C, for 1 h. Allow it to cool to ambient temperature in a desiccator and weigh it to the nearest 0,001 g. Place it in a funnel mounted on a second conical flask (**5.1**).

When dissolution of the soap appears to be complete, decant the supernatant liquid on to the filter paper, wash the insoluble matter in the conical flask by decantation with the ethanol (**4.1**), previously heated to near its boiling point, and transfer the insoluble matter to the filter paper with the aid of small quantities of warm ethanol (**4.1**).

Wash the filter paper and the residue with the warm ethanol until entirely free from soap.[1)]

During this operation, it is advantageous to place the conical flask carrying the funnel on the water bath (**5.3**) so as to keep the flitrate gently boiling. An independently heated funnel may also be used.

At the same time, cover the funnel with a watch glass. By this means, cooling of the wash liquor is avoided and ethanol vapour, which condenses on the watch glass and drops back on the filter paper completes the washing of the latter. Dry the filter paper in air and then place it in the oven (**5.4**), controlled at 103 ± 2 °C. After 1 h, remove the filter paper, leave it in the desiccator just long enough for it to cool completely to ambient temperature and weigh it. Repeat the operations of drying in the oven, cooling and weighing until the difference in mass between two successive weighings is less than 0,001 g. Note the final mass.

NOTE With certain soaps, especially silicated soaps, the insoluble matter cannot be completely detached from the bottom of the conical flask. In this case, after thoroughly washing the residue with ethanol, dissolve it in a little hot distilled water. Transfer this solution to a weighed evaporating dish, evaporate on the boiling water bath and dry in the oven (**5.4**), controlled at 103 ± 2 °C. Allow to cool in a desiccator and weigh. Repeat the operations of drying (for periods of 1 h), cooling and weighing until the difference in mass between two successive weighings is less than 0,001 g. Add this mass to that of the residue on the filter paper.

[1)] The final washings should show no appreciable residue on evaporation.

8 Expression of results

8.1 Calculation

The content of ethanol-insoluble matter, expressed as a percentage by mass, is given by the formula

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

where

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

m is the mass, in grams, of the residue.

8.2 Reproducibility

The difference between the results obtained on the same sample in two different laboratories shall not exceed 0,05 % for contents of ethanol-insoluble matter less than or equal to 1 %, and shall not exceed 0,1 % for contents of ethanol-insoluble matter higher than 1 %.

9 Test report

The test report shall include the following particulars:

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

b) the reference of the method used (reference to this International Standard);

c) the results, together with the form in which they are expressed;

d) the test conditions;

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

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