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BRITISH STANDARD

BS 1715-2.8: 1989
ISO 1066:1975

Analysis of soaps —

Part 2: Quantitative test methods —

Section 2.8 Method for determination of glycerol content

[ISO title: Analysis of soaps — Determination of glycerol content — Titrimetric method]

UDC 661.18:543

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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
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

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National foreword

This Section of BS 1715 has been prepared under the direction of the Chemicals Standards Policy Committee. It is identical with ISO 1066:1975 "*Analysis of soaps — Determination of glycerol content — Titrimetric method*" published by the International Organization for Standardization (ISO).

This method supersedes the methods "*Determination of glycerol*" 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 references

International Standard	Corresponding British Standard
ISO 1042:1983 (replacing ISO/R 1042)	BS 1792:1982 *Specification for one-mark volumetric flasks* (Identical)
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 **6.4** and **6.6**, British Standards related to ISO/R 385 (now revised as ISO 385-1:1984, ISO 385-2:1984 and ISO 385-3:1984) and ISO/R 648 (now revised as ISO 648:1977) are BS 846:1985 "*Specification for burettes*" and BS 1583:1986 "*Specification for one-mark pipettes*", respectively.

There is no British Standard corresponding to ISO 2272 but, as this reference is supplied only for information, the validity of this British Standard is not affected.

The International Standard for sampling of soaps (see clause **3** 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 **5.4**, the sulphuric acid solution, expressed as an amount-of-substance concentration, is $c(H_2SO_4)$ = 3.5 mol/L. With reference to **5.5**, **5.6** and **5.7**, the sodium hydroxide solutions, expressed as amount-of-substance concentrations, are c(NaOH) = 2 mol/L, c(NaOH) = 0.05 mol/L and c(NaOH) = 0.125 mol/L respectively.

With reference to **5.8**, the sulphuric acid solution, expressed as an amount-of-substance concentration, is $c(H_2SO_4)$ = 0.05 mol/L.

Also, in the UK it is considered that sodium periodate having a minimum purity of 99.0 % (m/m) is adequate.

In the definition of T in **9.1**, "normality" should be read as "amount-of-substance concentration (mol/L)".

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 1715-2.8.

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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.

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1 Scope

This International Standard specifies a titrimetric method for the determination of the glycerol content of commercial soaps, excluding compounded products.

2 Field of application

This method is applicable to soaps with glycerol contents equal to or greater than 0,5 % (*m*/*m*)[1]. This method is not applicable in the presence of organic compounds containing more than two hydroxyl groups on adjacent carbon atoms.

3 References

ISO 2272, *Surface active agents — Analysis of soaps — Determination of low contents of free glycerol — Spectrophotometric method.*

ISO ..., *Soaps — Sampling*[2].

4 Principle

Decomposition of the soap with sulphuric acid, and extraction of the fatty acids with light petroleum. Oxidation of the glycerol by periodic acid to formic acid and formaldehyde, and titration of the formic acid produced, using a pH meter.

5 Reagents

The reagents used shall be of recognized analytical purity and shall have the following properties.

5.1 *Distilled water*, from which carbon dioxide has been removed by boiling for 15 min and cooling in a vessel protected from atmospheric carbon dioxide.

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

5.3 *1,2-Ethanediol*, 50 % (*V*/*V*) aqueous solution.

5.4 *Sulphuric acid*, approximately 7 N solution.

5.5 *Sodium hydroxide*, 2 N solution.

5.6 *Sodium hydroxide*, 0,05 N solution.

5.7 *Sodium hydroxide*, 0,125 N standard volumetric solution, carbonate free.

5.8 *Sodium periodate* solution, prepared as follows:

Dissolve, at room temperature, 60 ± 0,5 g of sodium periodate ($NaIO_4$), minimum purity 99,8 %, in distilled water containing 120 ml of approximately 0,1 N sulphuric acid solution. Dilute to 1 l.

If the solution is turbid, filter it through a glass filter of porosity 16 to 40 μm, and place it in a brown glass bottle, which should be kept stoppered and in the dark.

6 Apparatus

Ordinary laboratory apparatus, and in particular:

6.1 *Beakers*, capacity 250 and 600 ml.

6.2 *Separating funnels*, capacity 250 ml.

6.3 *One-mark volumetric flasks*, capacity 250 ml, complying with the requirements of class A of ISO/R 1042.

6.4 *Burette*, capacity 50 ml, complying with the requirements of class A of ISO/R 385. The drainage time shall be not less than 90 s for 50 ml.

6.5 *Flat-bottomed or round-bottomed flask*, capacity 500 ml, complying with the requirements of ISO/R 1773.

6.6 *Pipette*, capacity 50 ml, complying with the requirements of class A of ISO/R 648, with a stated drainage time in order to ensure delivery of a constant volume.

6.7 *Variable-speed stirrer* (preferably magnetic) with glass paddles.

6.8 *pH meter* fitted with a glass electrode.

The pH meter shall be calibrated by means of two standard buffer solutions:

— potassium hydrogen phthalate $[C_6H_4(COOK)\,(COOH)]$, 0,05 M solution, pH 4,00 at 20 °C;

— disodium tetraborate decahydrate $[Na_2B_4O_7 \cdot 10H_2O]$, 0,01 M solution, pH 9,22 at 20 °C.

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 0,01 g, about 10 g of the laboratory sample into a 250 ml beaker (**6.1**).

8.2 Blank test

Carry out a blank test under the same conditions as the test itself, without adding soap, with 100 ml of the water (**5.1**). Adjust the pH to 8,1 ± 0,1, as specified in **8.5**, but carry out the final titration to pH 6,5 ± 0,1.

[1] For a glycerol content of less than 0,5 % (*m*/*m*), the method specified in ISO 2272 should be used.
[2] In preparation.

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8.3 Decomposition of soap and removal of fatty acids

Dissolve the test portion in 100 ml of the hot water (**5.1**). When dissolution is complete, transfer quantitatively into a separating funnel (**6.2**), rinsing the beaker with a little of the water.

Add about 10 ml of the sulphuric acid solution (**5.4**), shake, and allow to cool. After cooling, add about 100 ml of the light petroleum (**5.2**), shake, and allow the layers to separate. Draw off the aqueous layer into a second separating funnel, extract it again with about 50 ml of the light petroleum, shake and allow to settle.

Draw off the aqueous layer into a third separating funnel, extract it a third time with 50 ml of the light petroleum, shake, and allow to settle. Draw off the aqueous layer into a 250 ml one-mark volumetric flask (**6.3**).

Combine the ethereal extracts and wash them twice, each time with about 50 ml of the water.

Combine the washings with the acid solution in the 250 ml one-mark volumetric flask and dilute to the mark with the water (**5.1**).

8.4 Aliquot portion to be taken for determination

The best conditions for the determination of glycerol using 50 ml of the sodium periodate solution (**5.8**) are obtained when the aliquot portion on which the test is to be carried out contains between 0,3 and 0,5 g of glycerol. The volume of the aliquot portion be taken for the determination is shown in the following table:

Expected glycerol content in laboratory sample	Volume of acid solution to be taken for determination (from the 250 ml flask)
% *(m/m)*	ml
16 to 20	50
12 to 16	75
8 to 12	100
6 to 8	150
4 to 6	200
less than 4	250

If the amount taken for determination is less than recommended, the results obtained will be too high and lack precision. If the amount taken for determination is greater than that recommended, the results obtained will be low. If the result obtained does not fall within the expected range, as set out in the table, repeat the determination on another aliquot portion. For glycerol contents less than 2,5 % (*m*/*m*), reduce the volume of the sodium periodate solution to 25 ml.

8.5 Determination

Introduce the aliquot portion to be tested into the 500 ml flask (**6.5**). Boil[3)] the solution gently for 5 min to expel any carbon dioxide and light petroleum that may be present. Allow to cool with a carbon dioxide trap in the neck of the flask. Transfer the aliquot portion quantitatively into a 600 ml beaker (**6.1**). If the volume of liquid in the beaker is too small to permit adequate stirring, add a sufficient quantity of the water (**5.1**). If the pH of the solution is less than 3, insert the glass electrode (**6.8**) and start the stirrer (**6.7**). Add, drop by drop, the 2 N sodium hydroxide solution (**5.5**) until the pH is 3, then add, drop by drop, the 0,05 N sodium hydroxide solution (**5.6**) until the pH is 8,1 ± 0,1.

Pipette exactly 50 ml (or 25 ml, as appropriate) of the sodium periodate solution (**5.8**) into the solution. Mix the solution thoroughly with the aid of the stirrer, cover the flask with a watch-glass and leave for 30 min in the dark at room temperature (below 35 °C). When 30 min have elapsed, add 10 ml of the 1,2-ethanediol solution (**5.3**) and mix well. Cover again with the watch-glass and leave for 20 min in the dark at room temperature (below 35 °C).

Then titrate, using the pH meter (**6.8**), with the 0,125 N sodium hydroxide solution (**5.7**) until a pH of 8,1 ± 0,1 is reached. Note the volume used, to the nearest 0,05 ml.

9 Expression of results

9.1 Method of calculation

The glycerol content, is given, as a percentage by mass, by the formula

$$0,092\,1 \times (V_1 - V_2) \times T \times \frac{250}{V_0} \times \frac{100}{m}$$

$$= \frac{2\,302 \times T \times (V_1 - V_2)}{V_0 \times m}$$

where

V_0 is the volume, in millilitres, of the aliquot portion taken for the determination;

V_1 is the volume, in millilitres, of the 0,125 N sodium hydroxide solution (**5.7**) used for the determination;

V_2 is the volume, in millilitres, of the 0,125 N sodium hydroxide solution (**5.7**) used in the blank test;

T is the exact normality of the 0,125 N sodium hydroxide solution (**5.7**);

m is the mass, in grams, of the test portion.

[3)] A naked flame must not be used for heating because of the risk of combustion of flammable gases.

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9.2 Repeatability

The maximum difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst, in tests carried out by 23 laboratories on a sample containing 2,7 % (*m*/*m*) of glycerol, was 0,04 %.

9.3 Reproducibility

The maximum difference between the results of two tests in different laboratories on the sample containing 2,7 % (*m*/*m*) of glycerol, in tests carried out by 23 laboratories, was 0,2 %.

10 Test report

The test report shall include the following particulars:

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

b) the method used;

c) the results obtained;

d) the test conditions;

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

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Publications referred to

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