

Analysis of surface active agents (raw materials) —

Part 5: Ethoxylated alcohol and alkylphenol sulphates —

Section 5.2 Method for estimation of mean relative molecular mass

[ISO title: Surface active agents — Sulfated ethoxylated alcohols
and alkylphenols — Estimation of the mean relative molecular
mass]

UDC 661.185.1:543.2 – 112

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Chemicals Standards 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)
 London Regional Transport
 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 Committee, was published under the authority of the Board of BSI and comes into effect on 31 March 1989

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The following BSI references relate to the work on this standard:
 Committee reference CIC/34
 Draft for comment 86/50631 DC

ISBN 0 580 17137 X

Amendments issued since publication

Amd. No.	Date of issue	Comments

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National foreword

This Section of BS 6829 has been prepared under the direction of the Chemicals Standards Committee. It is identical with ISO 6843:1988 “*Surface active agents — Sulfated ethoxylated alcohols and alkylphenols — Estimation of the mean relative molecular mass*”, published by the International Organization for Standardization (ISO).

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”.

In British Standards it is current practice to use the spelling “sulphur”, etc., instead of “sulfur”, etc.

Wherever the words “International Standard” appear, referring to this Standard, they should be read as “Section of BS 6829”.

Cross-references

International Standard	Corresponding British Standard
ISO 607:1980	BS 3762 <i>Analysis of formulated detergents</i> Part 1:1983 <i>Methods of sample division</i> (Identical)
ISO 1042:1983	BS 1792:1982 <i>Specification for one-mark volumetric flasks</i> (Identical)
ISO 2271:1972	BS 3762 <i>Analysis of formulated detergents</i> Part 3: <i>Quantitative test methods</i> Section 3.1:1983 <i>Method for determination of anionic-active matter content</i> (Identical)
ISO 4800:1977	BS 2021:1980 <i>Specification for separating and dropping funnels for laboratory use</i> (Identical) BS 6829 <i>Analysis of surface active agents (raw materials)</i> Part 5: <i>Ethoxylated alcohol and alkylphenol sulphates</i> Section 5.1:1987 <i>Method for determination of total active matter content</i> (Identical)
ISO 6842:1983	Section 5.3:1988 <i>Method for determination of unsulphated matter content</i> (Identical)

Additional information. With reference to clause 4, water complying with grade 3 of BS 3978 “*Specification for water for laboratory use*” is suitable. With reference to 4.5, the concentration of the hydrochloric acid solution required is $c(\text{HCl}) = 2 \text{ mol/L}$.

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 6829-5.2.

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 to 6, 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.

1 Scope

This International Standard specifies a method for the estimation of the mean relative molecular mass of the anionic-active matter present in ordinary commercial neutralized products of sulfation of ethoxylated alcohols or alkylphenols [alkyl oxyethylene sulfates (ethoxylated alcohol sulfates) or alkylphenol oxyethylene sulfates (ethoxylated alkylphenol sulfates)] containing an average of not more than 20 oxyethylene groups per molecule.

It also sets out, in Annex A, a general scheme of analysis.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 607:1980, *Surface active agents and detergents — Methods of sample division*.

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks*.

ISO 2271:1972, *Surface active agents — Detergents — Determination of anionic-active matter (Direct two-phase titration procedure)*.

ISO 4800:1977, *Laboratory glassware — Separating funnels and dropping funnels*.

ISO 6842:1983, *Surface active agents — Polyethoxylated alcohol and alkylphenol sulfates — Determination of total active matter*.

ISO 8799:1988, *Sulfated ethoxylated alcohols and alkylphenols — Determination of content of unsulfated matter*.

3 Principle

From a solution of the test portion saturated with sodium chloride, extraction of alkylether sulfate with an ethyl acetate/butan-1-ol mixture, then evaporation of the aqueous phase containing the polyglycol, the polyglycol sulfate and possibly traces of ether sulfates; then removal of salts from the residue by treatment with methanol and filtration.

Evaporation of an aliquot portion of the filtrate and weighing of the residue, then redissolution in water and determination of the sodium chloride and anionic-active matter contents.

Determination of the polyglycol content by passing the remaining fraction of filtrate through an ion-exchange resin.

Determination of the polyglycol sulfate content by difference between the above determinations.

From the content of anionic surface active agent (alkylether sulfate) and the anionic surface active matter content determined by two-phase titration, estimation of the mean relative molecular mass.

NOTE The content of anionic surface active agent (alkylether sulfate) is obtained by the difference between the total active matter content and the unsulfated matter and polyglycol sulfate contents.

4 Reagents and materials

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

4.1 Methanol

4.2 Sodium chloride

4.3 *Ethyl acetate/butan-1-ol*, mixture (9 + 1) by volume.

4.4 *Sodium chloride*, 59 g/l solution.

4.5 *Hydrochloric acid*, 73 g/l solution.

4.6 *Sodium hydroxide*, 80 g/l solution.

4.7 *Silver nitrate*, standard volumetric solution, $c(\text{AgNO}_3) = 0,100 \text{ mol/l}$.

4.8 *Cation-exchange resin*, polystyrene sulfonic acid type, 2 % crosslinked, 150 to 330 μm , hydrogen form.

4.9 *Anion-exchange resin*, polystyrene quaternary ammonium type, 2 % crosslinked, 150 to 330 μm , chloride form.

5 Apparatus

Ordinary laboratory apparatus and

5.1 *Separating funnel*, of capacity 250 ml, in accordance with ISO 4800.

5.2 *Separating funnel*, jacketed, of capacity 250 ml.

5.3 *One-mark volumetric flask*, of capacity 100 ml, in accordance with ISO 1042.

5.4 *Water bath*, capable of being controlled at 50 °C.

5.5 *Reflux condenser*

5.6 *Rotary evaporator*, with round-bottom *flasks* of capacity 250 ml.

5.7 *Ion-exchange column*: Glass tube of internal diameter 25 mm and length 200 mm, restricted at the bottom and provided with a glass stopcock.

The ion-exchange resin is supported at the bottom by a 10 to 20 mm layer of glass wool or by a sintered glass filter.

6 Sampling

The laboratory sample of surface active agent shall be prepared and stored in accordance with the instructions given in ISO 607.

7 Procedure

7.1 Test portion

From the laboratory sample, if necessary homogenized by introducing a definite and appropriate quantity of water, weigh, to the nearest 0,1 g, into a 100 ml beaker, a quantity corresponding to about 25 mmol of anionic-active matter.

7.2 Preparation of the ion-exchange resins

If preferred, the resins may be prepared in smaller amounts than 1 kg with proportionately smaller volumes of reagents.

7.2.1 Anion-exchange resin

Take 1 kg of the anion-exchange resin (4.9) and allow to distend in water for 48 h. Transfer the resin to a suitable column and pass 5 litres of the sodium hydroxide solution (4.6) through the column, followed by 2 to 3 litres of water. Then pass 4 litres of the hydrochloric acid solution (4.5) through the column and again wash with 2 to 3 litres of water. The treated resin may be stored in water.

7.2.2 Cation-exchange resin

Take 1 kg of the cation-exchange resin (4.8) and allow to distend in water for 48 h. Transfer the resin to a suitable column and pass 5 litres of the hydrochloric acid solution (4.5) through the column, then wash with water until the washings are neutral. The treated resin may be stored in water.

7.3 Final preparation of resins

Take the required amount of anion-exchange resin, prepared as specified in 7.2.1, namely 25 ml per determination, and transfer to a suitable column. Pass through the column five times its volume of the sodium hydroxide solution (4.6), wash with water until neutral, then wash with 1 to 2 volumes of the methanol (4.1).

Take the required amount (25 ml) of cation-exchange resin, prepared as specified in 7.2.2, and, after introduction into a suitable column, wash with twice its volume of methanol (4.1).

7.4 Arrangement of the mixed-bed exchange column

Mix 25 ml of the cation-exchange resin and 25 ml of the anion-exchange resin, prepared as specified in 7.3, in a beaker. Fill the column (5.7) with the mixed resin in small portions and compress the mixed resin with a glass rod to a volume between 50 and 60 ml, and wash with 500 ml of the methanol (4.1).

7.5 Separation of polyglycol sulfate

7.5.1 Dissolve the test portion (7.1) in 50 ml of the sodium chloride solution (4.4) and 50 ml of the ethyl acetate/butan-1-ol mixture (4.3).

Transfer the solution to a separating funnel (5.1) and rinse the beaker with a few millilitres of the sodium chloride solution (4.4) and of the ethyl acetate/butan-1-ol mixture, then add the rinsing solution to the separating funnel.

Shake the separating funnel vigorously and allow the phases to separate.

Draw off the aqueous phase into the jacketed separating funnel (5.2), thermoregulated at 60 °C, add 5 g of the sodium chloride (4.2) and shake to complete dissolution. Add 50 ml of the ethyl acetate/butan-1-ol mixture and shake.

After separation of the phases, draw off the aqueous phase into a 250 ml round-bottom flask (5.6) and evaporate to dryness by means of the rotary evaporator (5.6) on a water bath (5.4), controlled at 50 °C, under vacuum, using a water-jet air pump.

Add to the residue 30 ml of the methanol (4.1) and boil under reflux for 3 min and filter through a medium-speed filter paper, collecting the filtrate in a 250 ml flask (5.6). Repeat these operations three times. Evaporate to dryness using the rotary evaporator.

Treat the new residue with 30 ml of the methanol (4.1), boil under reflux for 3 min and filter through a medium-speed filter paper, collecting the filtrate in the 100 ml one-mark volumetric flask (5.3). Repeat these operations three times and dilute to the mark with the methanol.

By means of a pipette, transfer 50 ml of the solution to a tared 150 ml beaker, evaporate under a nitrogen current, dry at 105 °C to constant mass, and weigh the residue (m_1).

7.5.2 Pass the 50 ml remaining in the volumetric flask through the column prepared as specified in 7.4.

Regulate the rate of flow to 5 ml/min and wash the column with 100 ml of the methanol. Transfer the eluate to a tared 250 ml round-bottom flask (5.6) and evaporate with the rotary evaporator on the water bath, controlled at 50 °C, under vacuum, using a water-jet air pump. Dry at 105 °C to constant mass, and weigh the residue (m_2).

7.5.3 Dissolve the dry residue (m_1) in 50 ml of water. On 10 ml of this solution, determine the anionic-active matter content (A_2) by direct two-phase titration in accordance with ISO 2271.

7.5.4 On another 10 ml of this solution, determine the chloride content by titration with the silver nitrate solution (4.7).

7.6 Estimation of the mean relative molecular mass

The mean relative molecular mass is obtained by calculation (see 8.1.2).

8 Expression of results

8.1 Method of calculation

8.1.1 Polyglycol sulfate content

The polyglycol sulfate content PS, expressed as a percentage by mass, is given by the equation

$$PS = R - P - A$$

where the meaning of the letter symbols is as follows.

8.1.1.1 R is the percentage by mass of residue in the aqueous phase after extraction by the ethyl acetate/butan-1-ol mixture, given by the equation

$$R = \frac{(2 m_1 - 0,058 5 V) \times 100}{m_0}$$

where

m_0 is the mass, in grams, of the test portion (7.1);

m_1 is the mass, in grams, of the residue from the aqueous phase obtained in 7.5.1;

V is the volume, in millilitres, of the silver nitrate solution (4.7) used in 7.5.4.

8.1.1.2 P is the percentage by mass of polyglycol in the aqueous phase after extraction by the ethyl acetate/butan-1-ol mixture, given by the equation

$$P = \frac{2 m_2 \times 100}{m_0}$$

where

m_0 is the mass, in grams, of the test portion (7.1);

m_2 is the mass, in grams, of the residue obtained after passing 50 ml of the methanolic solution prepared in 7.5.1 through the ion-exchange column as described in 7.5.2.

8.1.1.3 A is the percentage by mass of anionic-active matter in the aqueous phase after extraction by the ethyl acetate/butan-1-ol mixture, given by the equation

$$A = \frac{A_2 (AT + P - NS - R)}{A_1 - A_2}$$

where

A_1 is the anionic-active matter content, expressed in millimoles per gram, determined on the sample by direct two-phase titration in accordance with ISO 2271;

A_2 is the anionic-active matter content, expressed in millimoles per gram, determined on the aqueous phase after extraction by the ethyl acetate/butan-1-ol mixture (7.5.1) by direct two-phase titration (7.5.3);

AT is the total active matter content, expressed as a percentage by mass, determined on the sample in accordance with ISO 6842;

P is the polyglycol content, calculated in accordance with 8.1.1.2, expressed as a percentage by mass;

NS is the unsulfated matter content, expressed as a percentage by mass, determined on the sample in accordance with ISO 8799;

R is the percentage by mass of residue in the aqueous phase after extraction by the ethyl acetate/butan-1-ol mixture, calculated in accordance with 8.1.1.1.

8.1.2 Mean relative molecular mass

The mean relative molecular mass is given by the formula

$$10 \times \frac{(AT - NS - PS)}{A_1}$$

where

AT is the total active matter content, expressed as a percentage by mass, determined on the sample in accordance with ISO 6842;

NS is the unsulfated matter content, expressed as a percentage by mass, determined on the sample in accordance with ISO 8799;

PS is the polyglycol sulfate content, expressed as a percentage by mass, calculated in accordance with 8.1.1;

A₁ is the anionic-active matter content, expressed in millimoles per gram, determined on the sample by direct two-phase titration in accordance with ISO 2271.

8.2 Precision

8.2.1 Polyglycol sulfate content

Comparative analyses carried out in fifteen laboratories has given the following statistical results:

- mean [% (*m/m*) of polyglycolsulfate] : 1,08
- standard deviation of repeatability, σ_r : 0,18
- standard deviation of reproducibility, σ_R : 0,77

8.2.2 Mean relative molecular mass

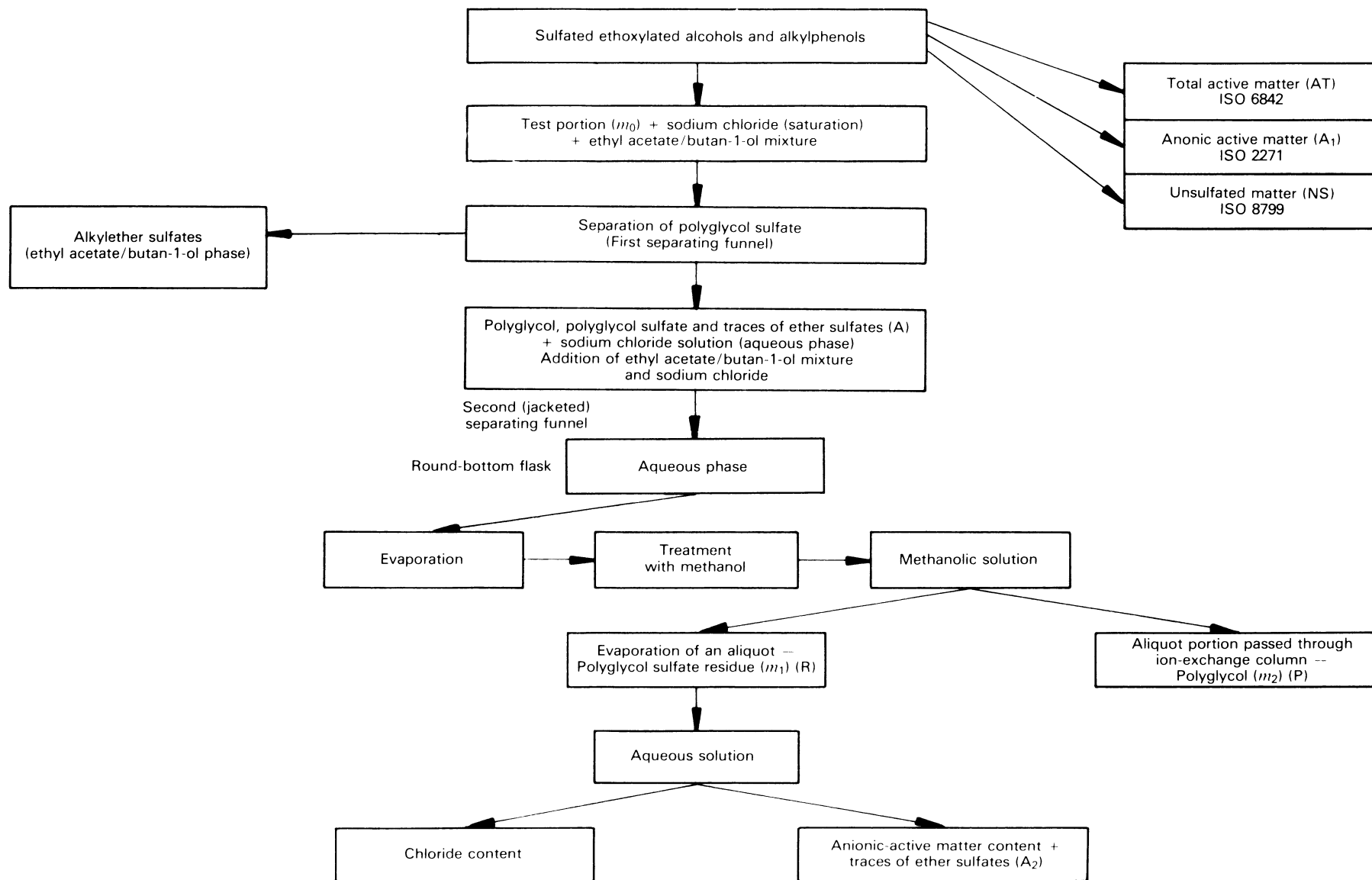
Because of the differences between the various methods used for the estimation and the calculation of the mean relative molecular mass, it is not possible to give statistical results on the validity of the estimation.

9 Test report

The test report shall include the following information:

- a) all information necessary for the complete identification of the sample;
- b) the method used (reference to this International Standard);
- c) the results and the method of expression used;
- d) any operational details not specified in this International Standard or in the International Standards to which reference is made, or any operational details regarded as optional, as well as any incidents liable to have affected the results.

Annex A (normative) General scheme of analysis



Publications referred to

See national foreword.

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