BRITISH STANDARD

BS 6829-5.1: 1990
ISO 6842:1989

Analysis of surface active agents (raw materials) —

Part 5: Ethoxylated alcohol and alkylphenol sulphates —

Section 5.1 Method for determination of total active matter content

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 January 1990

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Contents

National foreword

This Section of BS 6829 has been prepared under the direction of the Chemicals Standards Policy Committee. It is identical with ISO 6842:1989 "*Surface active agents — Sulfated ethoxylated alcohols and alkylphenols — Determination of total active matter content*", published by the International Organization for Standardization (ISO).

This Section of BS 6829 supersedes the 1987 edition of BS 6829-5.1, which is withdrawn. The principal difference between this Section of BS 6829 and the 1987 edition is the correction of an error in **8.1**, although minor editorial modifications have also been made.

Cross-reference

International Standard	Corresponding British Standard
ISO 607:1980	BS 3762 *Analysis of formulated detergents* Part 1:1983 *Methods of sample division* (Identical)

Additional information. With reference to clause **4**, water complying with grade 3 of BS 3978 "*Specification for water for laboratory use*" is suitable.

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 6829-5.1.

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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, 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 determination of the total 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)].

The total active matter comprises the organic material soluble in ethanol (alkylether sulfates, alkylphenylether sulfates, polyglycol sulfates and non-ionic fractions).

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was 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 edition of the standard indicated 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.*

3 Principle

Boiling, under reflux, of a test portion with ethanol in the presence of sodium sulfate. Filtration, evaporation of the filtrate and weighing of the residue. Determination of any sodium chloride present, by dissolution of the residue in aqueous acetone and titration with standard volumetric silver nitrate solution. Correction of the mass of the residue for the sodium chloride content.

4 Reagents

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

4.1 *Ethanol,* 99 % (*V*/*V*).

4.2 *Dichloromethane*

4.3 *Sodium sulfate,* anhydrous.

4.4 *Acetone,* 50 % (*V*/*V*) aqueous solution.

4.5 *Silver nitrate,* standard volumetric solution, $c(AgNO_3)$ = 0,1 mol/l.

4.6 *Potassium chromate,* 100 g/l indicator solution.

5 Apparatus

Ordinary laboratory apparatus and:

5.1 *Conical flask,* of capacity 250 ml, with a ground glass neck.

5.2 *Rotary evaporator,* with *round-bottomed flasks* of capacity 250 ml.

5.3 *Condenser,* to fit the conical flask (**5.1**).

6 Sampling

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

7 Procedure

7.1 Test portion

From the laboratory sample, rendered homogeneous (if necessary) by the addition of a known, appropriate quantity of water, weigh, to the nearest 1 mg, into the conical flask (**5.1**) a quantity of homogeneous material containing about 0,5 g to 1,5 g of total active matter.

7.2 Determination

Introduce into the conical flask containing the test portion (**7.1**) 100 ml of ethanol (**4.1**) and 100 mg of sodium sulfate (**4.3**), fit the condenser (**5.3**), and boil under reflux for 30 min.

Disconnect the condenser. Rinse the inner wall of the condenser and the neck of the flask with ethanol, collecting the washings in the flask. Allow to settle.

Filter the contents of the conical flask whilst still hot through a fast filter paper into one of the round-bottomed flasks (**5.2**), previously dried and tared to the nearest 1 mg. Rinse the conical flask with about 50 ml of hot ethanol, filtering the washings into the round-bottomed flask.

Evaporate the ethanolic solution by means of the rotary evaporator (**5.2**) maintained at approximately 40 °C. Add 10 ml of dichloromethane (**4.2**) and evaporate. Repeat this step using a further 10 ml of dichloromethane. Remove the last traces of water by evaporation and leave the flask for a further 15 min on the rotary evaporator.

Remove the flask from the rotary evaporator, allow to stand in a desiccator for 15 min and weigh the flask and contents.

Place the flask on the rotary evaporator for a further 15 min, then allow it to stand in the desiccator for 15 min and again weigh the flask and contents. Repeat the process of drying and weighing until the difference between two consecutive weighings does not exceed 3 mg.

Dissolve the residue in 60 ml to 80 ml of the aqueous acetone (**4.4**). Add 1 ml of the potassium chromate indicator solution (**4.6**) and titrate with the silver nitrate solution (**4.5**) until a permanent brown coloration is obtained.

7.3 Blank test

Carry out a blank test at the same time as the determination, using the same reagents and following the same procedure, but omitting the test portion.

8 Expression of results

8.1 Method of calculation

The total active matter content, expressed as a percentage by mass, is given by the formula

$$\frac{m_1 - 0{,}058\,5\,c(V_1 - V_0)}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion (**7.1**);[1)]

m_1 is the mass, in grams, of the residue obtained;

c is the actual concentration, in moles of $AgNO_3$ per litre, of the silver nitrate solution (**4.5**);

V_0 is the volume, in millilitres, of the silver nitrate solution (**4.5**) used for the blank test (**7.3**);

V_1 is the volume, in millilitres, of the silver nitrate solution (**4.5**) used for the determination (**7.2**) of any sodium chloride present;

0,058 5 is the mass, in grams, of sodium chloride corresponding to 1,00 ml of silver nitrate solution, $c(AgNO_3)$ = 1,000 mol/l.

8.2 Precision

Comparative analyses, carried out in 15 laboratories, have given the following statistical results:

— mean [total active matter, % (m/m)]: 58,67

— repeatability standard deviation, σ_r: 0,33

— reproducibility standard deviation, σ_R: 0,94

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 (a reference to this International Standard);

c) the results obtained and the units in which they have been expressed;

d) any operational details not specified in this International Standard or in the International Standard to which reference is made, and any operation regarded as optional, as well as any incidents likely to have affected the results.

[1)] Corrected for dilution in the case of heterogeneous products.

Publication referred to

See national foreword.

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