BRITISH STANDARD

BS EN
14670:2005

Surface active agents — Sodium dodecyl sulfate — Analytical method

The European Standard EN 14670:2005 has the status of a British Standard

ICS 71.100.40

BSI
British Standards

National foreword

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Summary of pages

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English version

Surface active agents - Sodium dodecyl sulfate - Analytical method

Agents de surface - Dodécylsulfate de sodium - Méthode d'analyse

Grenzflächenaktive Stoffe - Natriumdodecyulsulfat - Analysenverfahren

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Contents

Foreword

This European Standard (EN 14670:2005) has been prepared by Technical Committee CEN/TC 276 “Surface active agents”, the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 2005, and conflicting national standards shall be withdrawn at the latest by December 2005.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This European Standard specifies a method for the determination of the active content and molar mass of sodium dodecyl sulphate used as original titre for the titre determination of standard solutions for surface active agents titration.

NOTE These are used in the mixed indicator two-phase titration (e. g. ISO 2271) and in the potentiometric titration using a surface active agent sensitive electrode (e.g. EN 14480).

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 13267, *Surface active agents – Determination of water content – Karl Fischer method.*

EN ISO 3696, *Water for analytical laboratory use – Specification and test methods (ISO 3696:1987).*

ISO 607, *Surface active agents and detergents – Methods of sample division.*

ISO 894, *Surface active agents – Technical sodium primary alkylsulphates – Methods of analysis.*

ISO 6844, *Surface active agents – Determination of mineral sulfate content – Titrimetric method.*

3 Principle

The water content, the sodium sulphate content and the extractable matter content by light petroleum of the test sample are determined according to the methods respectively described in EN ISO 3696, ISO 894 and ISO 9844 and the active content is calculated by subtracting the water content, the sodium sulphate content and the content of extractable matter by light petroleum from 100 %. For calculating the average molar mass of the sodium dodecyl sulphate the fatty alcohols C_{10} to C_{14} released after acidic hydrolysis are isolated and the C-chain distribution is determined by gas chromatography.

4 Reagents

During the analysis, unless otherwise specified, use only reagents of recognized analytical grade which have been checked in advance as not interfering with the analytical results and water complying with grade 3 as defined in EN ISO 3696 and in particular use.

a) for the determination of the water content, the reagents specified in EN 13267;

b) for the determination of the sodium sulphate content, the reagents specified in ISO 6844 (or prEN 14880). ;

c) for the determination of extractable matter content by light petroleum, the reagents specified in ISO 894.

5 Apparatus

Normal laboratory apparatus and the following:

5.1 Gas chromatograph, suitable for work with a fused silica capillary column and equipped with a flame ionization detector (FID).

5.2 Capillary column, capable of the separation characteristics shown in Figure A.1.

NOTE A 30 m x 0.32 mm ID, fused silica capillary column (film thickness 0,25 µm) with 5 % phenyl-, 95 % methyl polysiloxane stationary phase is advisable.

6 Sampling and preparation of the sample

The laboratory sample shall be prepared and stored in accordance with ISO 607.

7 Procedure

7.1 Determination of water content

Determine the water content in accordance with EN 13267.

NOTE Usually the water content is < 1 % (m/m).

7.2 Determination of the sodium sulphate content

Determine the sodium sulphate content in accordance with ISO 6844 (dithizone method).

NOTE Usually the sodium sulphate content is < 0,1 % (m/m).

7.3 Determination of the extractable matter content by light petroleum

Determine the extractable matter content by light petroleum in accordance with ISO 894.

NOTE 1 Usually the content is < 0,2 % (m/m).

NOTE 2 For the determination of the unsulphated matter, EN 13273 may also be applied if an appropriate calibrant (C_{12}-alcohol) is available.

7.4 Determination of the average molar mass of sodium dodecyl sulfate

Extract the light petroleum soluble matter in accordance with ISO 894 from the neutralized aqueous ethanolic solution of the test sample.

After acid hydrolysis of the alkyl sulphate remaining in the aqueous ethanolic phase, extract the released fatty alcohol with diethyl ether in accordance with ISO 894.

Evaporate the dried diethyl ether extract to a volume of approximately 20 ml. Determine the C-chain distribution of the fatty alcohol using gas chromatography with the given parameters. The peak assignment can be done with a fatty alcohol kit serving as reference. At least the quality of separation shown in Figure A.1 shall be achieved.

8 Expression of results

8.1 Calculation of the active content

The active content, *w*, expressed as grams per 100 g, is calculated according to equation (1):

$$w = 100 - w_{H_20} - w_{Na_2SO_4} - w_{me} \quad (1)$$

where

w_{H_20} is the water content according to 7.1, in grams per 100 g;

$w_{Na_2SO_4}$ is the sodium sulphate content according to 7.2, in grams per 100 g;

w_{me} is the content of matter extractable by light petroleum according to 7.3, in grams per 100 g.

8.2 Calculation of the average molar mass

The average molar mass, M, expressed as grams per mole, is calculated according to equation (2):

$$M = \left[\sum \left(A_n \times n\right)\right] \times 12 + \left[2 \sum \left(A_n \times n\right) + 1\right] \times 1 + 119 \quad (2)$$

where

n is the C-number of the fatty alcohol;

A_n is the relative area for the fatty alcohol n.

9 Precision

9.1 General

Despite the sometimes considerable statistical variations during the analysis of minor constituents, the subtraction method offers the most reliable results for the analysis of the active content. As the contents of the minor constituents are generally < 0,1 % (m/m) for sodium sulphate and unsulphated matter and < 1 % (m/m) for water, high variation coefficients have only low effect on the final result. The same applies to the high variation coefficient for the C_{10} and C_{14} fatty alcohol analysis.

9.2 Repeatability limit

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will not exceed the repeatability limit, *r*, in more than 5 % of cases.

Precision data are given in Annex B.

9.3 Reproducibility limit

The absolute difference between two independent single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, will not exceed the reproducibility limit, *R*, in more than 5 % of cases.

Precision data are given in Annex B.

10 Test report

The test report shall include the following information:

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

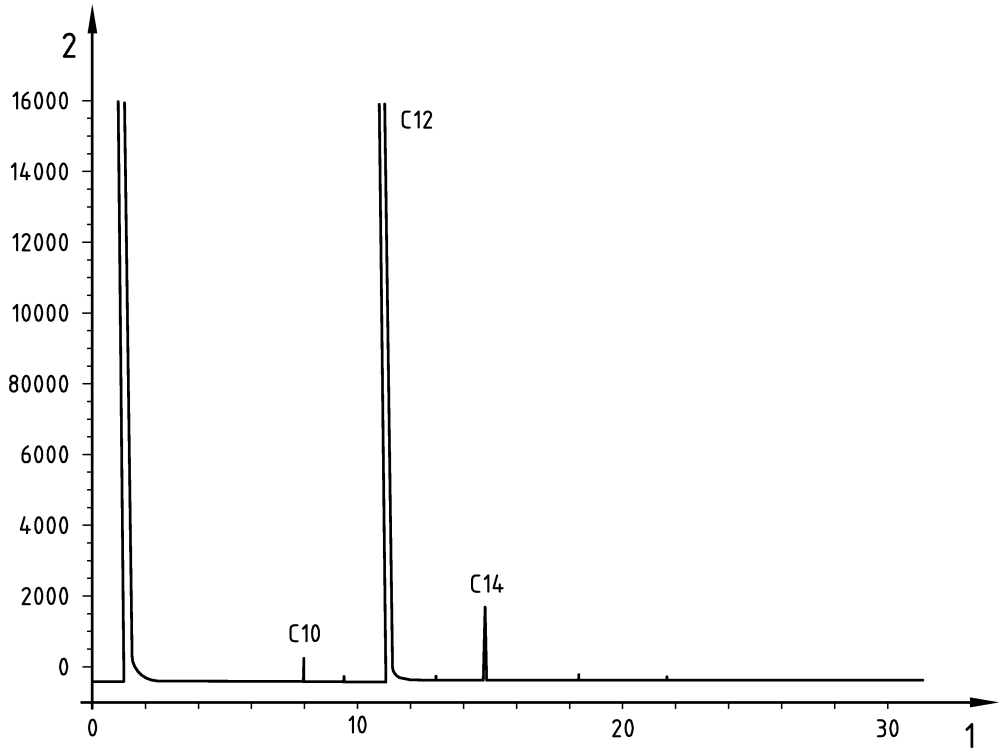
b) method used (a reference to this European Standard);

c) test results;

d) details of any operations not specified in this document or in the European standards to which reference is made, and any operations regarded as optional, as well as any incidents likely to have affected the results.

Annex A
(informative)

Typical gas chromatogram



Key

1 Retention time in minutes
2 Response, in millivolts

Figure A.1 - Typical gas chromatogram

The following gas chromatography conditions have been found to be suitable:

Column : A 30 m x 0.32 mm ID, fused silica capillary column (film thickness 0,25 µm) with 5 % phenyl-, 95 % methyl polysiloxane stationary phase is advisable.

Carrier gas:	helium
Column Head-pressure:	115 kPa
Gas flow:	3 ml/min
Split ratio:	1:50
Sample quantity:	1 µl of a 10 % solution
Temperature program:	50 °C for 1 min, then up to 120 °C at 15 °C/min, then up to 330 °C at 60 °C/min
Injector:	280 °C
Detector:	350 °C

Evaluation : normalisation of the peak area ratio to 100 % (GC area-percent)

Annex B
(informative)

Results of inter-laboratory test

The inter-laboratory test was carried out in 1992 by GAT (Gemeinschaftsausschuss für die Analytik von Tensiden). The results of the inter-laboratory test were evaluated in accordance with ISO 5725-2.

Table B.1 — Results of inter-laboratory test (active content)

Designation	Water	Na_2SO_4	Matter extractable by light petroleum	Active content
Method	EN 13267	ISO 6844	ISO 894	
Number of laboratories participating	10	10	10	10
Number of laboratories not eliminated	9	10	9	8
Number of individual test results of all laboratories	20	20	20	20
Mean value, *m*, in g/100 g	0,88	0,03	0,09	99,0
Repeatability standard deviation, S_r, in g/100 g	0,02	0,01	0,02	0,04
Repeatability, $r = (S_r \times 2{,}8)$, in g/100 g	0,06	0,03	0,06	0,12
Reproducibility standard deviation, S_R, in g/100 g	0,12	0,02	0,05	0,13
Reproducibility, $R = (S_R \times 2{,}8)$, in g/100 g	0,33	0,05	0,15	0,36
Repeatability coefficient of variation in %	2,3	33,3	22,2	0,04
Reproducibility coefficient of variation in %	13,6	66,6	55,6	0,13

Table B.2 — Results of inter-laboratory test (Gas chromatography data for molar mass determination)

Designation	C_{10}-OH	C_{12}-OH	C_{14}-OH
Number of laboratories participating	7	7	7
Number of laboratories not eliminated	5	7	6
Number of individual test results of all laboratories	10	14	14
Mean value, *m*, in g/100 g	0,2	99,4	0,6
Repeatability standard deviation, S_r, in g/100 g	0,03	0,07	0,07
Repeatability, $r = (S_r \times 2{,}8)$, in g/100 g	0,09	0,20	0,20
Reproducibility standard deviation, S_R, in g/100 g	0,07	0,25	0,09
Reproducibility, $R = (S_R \times 2{,}8)$, in g/100 g	0,21	0,71	0,26
Repeatability coefficient of variation in %	59,0	0,2	34,9
Reproducibility coefficient of variation in %	138,4	0,7	44,7

The average molar mass of the sodium dodecyl sulfate used in the inter-laboratory test is calculated according to the following equation based on the above results:

$$M = [0{,}002 \times 10 + 0{,}994 \times 12 + 0{,}006 \times 14]12 + [2(0{,}002 \times 10 + 0{,}994 \times 12 + 0{,}006 \times 14) + 1] \times 1 + 119$$

$$M = 288{,}4 \text{ g/mol}$$

Bibliography

[1] EN 13273, *Surface active agents – Determination of the content of non-ionic substances in anionic surface active agents by high performance liquid chromatography (HPLC).*

[2] EN 14480, *Surface active agents - Determination of anionic surface active agents - Potentiometric two-phase titration method*.

[3] prEN 14880, *Surface active agents - Determination of inorganic sulphate content in anionic surface active agents - Potentiometric lead selective electrode titration method*

[4] ISO 2271, *Surface active agents - Detergents - Determination of anionic-active matter by manual or mechanical direct two-phase titration procedure.*

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