BRITISH STANDARD

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13435:2002

Surface active agents — Determination of free amine content of alkyl dimethyl betaines

The European Standard EN 13435:2001 has the status of a British Standard

ICS 71.100.40

National foreword

This British Standard is the official English language version of EN 13435:2001.

The UK participation in its preparation was entrusted to Technical Committee CII/34, Methods of test for surface active agents, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

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This British Standard, having been prepared under the direction of the Materials and Chemicals Sector Policy and Strategy Committee, was published under the authority of the Standards Policy and Strategy Committee on 5 March 2002

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English version

Surface active agents - Determination of free amine content of alkyl dimethyl betaines

Agents de surface - Détermination de la teneur en amines libres dans les alkyl diméthylbétaïnes

Grenzflächenaktive Stoffe - Bestimmung des freien Amingehaltes von Alkyldimethylbetainen

This European Standard was approved by CEN on 11 November 2001.

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 276 "Agents de surface", 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of an identical text or by endorsement, at the latest by June 2002, and conflicting national standards shall be withdrawn at the latest by June 2002.

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

Annexes A, B and C are informative.

1 Scope

This European Standard specifies a method for the determination of 0,02 mmol to 1 mmol of free amine in alkyl dimethyl betaines. Monochloroacetic acid, glycolic acid and strong acids do not interfere the determination.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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*.

3 Term and definition

For the purposes of this European Standard, the following term and definition apply.

3.1
free amine
content of amine in alkyl dimethyl betaines which can be determined by this method, expressed in grams per 100 g

4 Principle

The sample of alkyl dimethyl betaine is dissolved in aqueous propan-2-ol. All the components are converted into their basic form by addition of sodium hydroxide solution. Carbon dioxide is removed by stripping out with nitrogen.

The free amine is determined by titration with hydrochloric acid using potentiometric indication of the end point.

NOTE In aqueous propan-2-ol the difference in basicity is sufficient for the free amine to be determined separately from any monochloroacetate, glycolate and betaine.

5 Reagents

During the analysis, unless otherwise specified, use only reagents of recognized analytical grade and have been checked in advance as to not interfere with the analytical results.

5.1 **Water**, complying with grade 3 as defined in EN ISO 3696.

5.2 **Hydrochloric acid standard volumetric solution**, *c*(HCl) = 0,1 mol/l.

5.3 **Sodium hydroxide solution**, *c*(NaOH) = 0,1 mol/l.

5.4 **Indicator solution**, thymol blue, 1 g/l in ethanol.

5.5 **Propan-2-ol/water mixture**, as volume fraction approximately 1 : 1.

5.6 **Tri-n-butylamine**, c($C_{12}H_{27}N$) = 0,02 mol/l in propan-2-ol.

5.7 **Acetic acid**, 100 % (glacial acetic acid).

5.8 **Nitrogen gas**, at least 99,9 %.

6 Apparatus

Ordinary laboratory apparatus and the following.

6.1 Potentiometric titrating apparatus, comprising a titrator with a combined glass/calomel electrode (c(LiCl) = 3 mol/l in ethanol), a piston burette of 20 ml capacity and a magnetic stirrer (see also Annex B).

6.2 Analytical balance, accurate to 0,1 mg.

7 Sampling and sample

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

Solid and pasty samples shall gently be melted on a water-bath at the lowest possible temperature (maximum 60 °C).

8 Procedure

8.1 Determination

Weigh approximately 10 g of the sample, to the nearest 1 mg, into a 250 ml glass beaker. Then dissolve the sample in 150 ml of propan-2-ol/water mixture (5.5).

Pass in nitrogen gas (5.8) for 5 min by means of a disposable glass pipette. Allow the tip of the pipette to dip approximately 2 cm into the sample solution and adjust the flow of nitrogen so that 2 to 3 bubbles emerge per second.

Add exactly 10 ml of tri-n-butylamine solution (5.6) and after five drops of indicator solution (5.4).

Add sodium hydroxide solution (5.3) dropwise until the indicator changes from yellow to blue and then a further millilitre of sodium hydroxide solution (5.3) in excess.

Immerse the electrode, stir and titrate with the hydrochloric acid standard volumetric solution (5.2) to the second inflexion point (see Figure C.1).

8.2 Blank determination

Determine the blank value according to the procedure given in 8.1. Use one drop of acetic acid (5.7) instead of the sample.

9 Expression of results

Calculate the content of free amine, *w,* expressed as grams per 100 g, according to the equation (1) :

$$w = \frac{[(V_1 - V_2) - (V_3 - V_4)] \times c \times M \times 100}{m \times 1000} \qquad (1)$$

where :

V_1 is the volume of hydrochloric acid standard volumetric solution (5.2) to the second inflexion point during titration of the sample, in millilitres ;

V_2 is the volume of hydrochloric acid standard volumetric solution (5.2) to the first inflexion point during titration of the sample, in millilitres ;

V_3 is the volume of hydrochloric acid standard volumetric solution (5.2) to the second inflexion point during titration of the blank test solution, in millilitres ;

V_4 is the volume of hydrochloric acid standard volumetric solution (5.2) to the first inflexion point during titration of the blank test solution, in millilitres ;

c is the amount-of-substance concentration of the hydrochloric acid standard volumetric solution (5.2), in moles per litre;

M is the molar mass of the free amine, in grams per mole ;

m is the mass of the sample, in grams.

The result shall be given to one decimal place.

10 Precision

10.1 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.

Typical precision data obtained in a ring test are given in annex A.

10.2 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.

Typical precision data obtained in a ring test are given in annex A.

11 Test report

The test report shall include the following information :

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

b) a reference to this European Standard ;

c) the results ;

d) any particular points observed in the course of the test ;

e) details of any operations not specified in this European Standard, and any operations regarded as optional, as well as any incidents likely to have affected the results.

Annex A
(informative)

Ring test results

The ring test was carried out by the German „Common Committee Analysis of Surfactants“ („Gemeinschaftsausschuss für die Analytik von Tensiden“).

The sample used was a commercially available alkyl dimethyl betaine (molar mass of the corresponding N,N-dimethylcocoamine: 226 g/mol).

The ring test results were evaluated in accordance with ISO 5725-2 (see Table A.1).

Table A.1 — Ring test results

Designation	Precision data
Number of laboratories participating	8
Number of laboratories not eliminated	8
Number of individual values of all laboratories	25
Mean value, w in g/100 g	0,24
Repeatability standard deviation, s_r in g/100 g	0,03
Repeatability limit, r ,(r = 2,8 x s_r) in g/100 g	0,08
Variation coefficient of the repeatability, in %	12,1
Reproducibility standard deviation, s_R in g/100 g	0,07
Reproducibility limit, R, (R = 2,8 x s_R) in g/100 g	0,19
Variation coefficient of the reproducibility, in %	29,3

Annex B
(informative)

Instrument settings

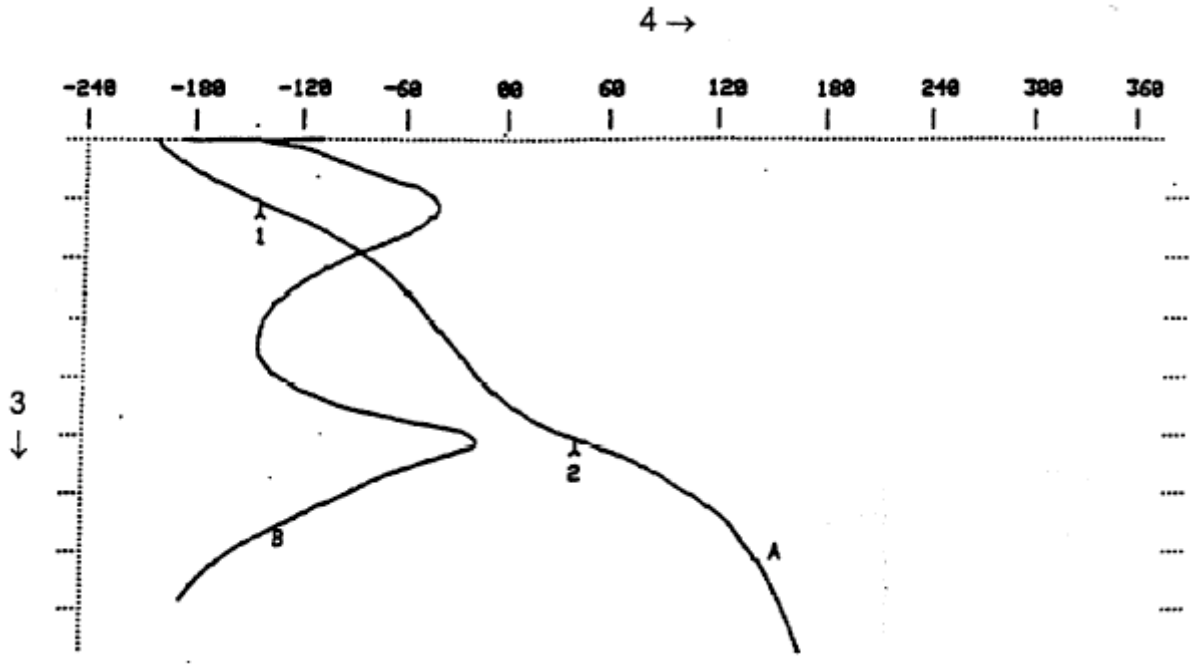
Data given as follows for instrument settings refer to the dynamic equivalence point titration. They are settings for the Metrohm Titroprocessor 670[1]) and are intended to act as a guideline, only :

Burette volume / Resolution:	20 ml / 2μl
Measured quantity:	V
Measuring point density:	4
Dosing rate for increments:	2 ml/min
Minimum increment on titration:	4 μl
Drift threshold for measured value acquisition:	100 mV/min
Equilibration time for measured value acquisition:	19 s

[1]) Metrohm Titroprocessor 670 is the trade name of an instrument supplied by Metrohm Ltd. (CH-9101 Herisau, Switzerland). This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of the instrument named.

Annex C
(informative)

Example for a typical titration curve and its derivative



Legend

1 Inflexion point 1
2 Inflexion point 2
3 Volume of hydrochloric acid standard volumetric solution in millilitres
4 Voltage in millivolts

Figure C.1 —Example of a typical titration curve (A) and its first derivative (B) of an alkyl dimethyl betaine

Bibliography

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

J.M. Plantinga, J.J. Donkerbroek and R. Mulder, JAOCS 70, 97 (1993)

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