BRITISH STANDARD

BS EN
14667:2005

Surface active agents — Determination of low boiling solvents in liquid formulations — Gas chromatographic method

The European Standard EN 14667:2005 has the status of a British Standard

ICS 71.100.40

BSI
British Standards

National foreword

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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:

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Summary of pages

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EUROPEAN STANDARD

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EN 14667

June 2005

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English version

Surface active agents - Determination of low boiling solvents in liquid formulations - Gas chromatographic method

Agents de surface - Détermination des solvants à faible point d'ébullition dans les formulations liquides - Méthode par chromatographie en phase gazeuse

Grenzflächenaktive Stoffe - Bestimmung des Gehaltes an leichtsiedenden Lösemitteln in flüssigen Formulierungen - Gaschromatographisches Verfahren

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Contents

Foreword

This European Standard (EN 14667:2005) 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of an identical text or by endorsement, at the latest by December 2005, and conflicting national standards shall be withdrawn at the latest by December 2005.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 content of low boiling solvents, e.g. methanol, ethanol propan-1-ol and propan-2-ol in liquid detergents. Typically only one solvent is present in these kinds of samples.

The method is applicable to liquid detergents containing low boiling solvents in the range from 0,5 % to 10 % by weight.

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.

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

3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply.

low boiling solvent
alcohol with a boiling point below 100 °C

NOTE For example ethanol, propan-1-ol and propan-2-ol.

4 Principle

The liquid detergent is dissolved in butan-1-ol, which is a water miscible solvent with higher boiling point than the solvents to be determined. The content of low boiling solvents is determined with gas chromatography.

5 Reagents

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

5.1 Ethanol, purity ≥ 99,5 % (m/m)

5.2 Propan-1-ol, purity ≥ 99,5 % (m/m).

5.3 Propan-2-ol, purity ≥ 99,5 % (m/m).

5.4 Butan-1-ol, purity ≥ 99,5 % (m/m).

5.5 Carrier gas, nitrogen or helium for gas chromatography.

5.6 Auxiliary gases, for gas chromatography.

6 Apparatus

Normal laboratory apparatus and the following:

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

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

NOTE A 30 m x 0,53 mm ID, fused silica capillary column (film thickness 0,25 µm) with 5 % phenyl-, 95 % methyl polysiloxane stationary phase is advisable.

6.3 Electronic integrator

6.4 Ultrasonic bath

6.5 Membrane filter, 0,45 µm pore size.

7 Sampling and preparation of the sample

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

8 Procedure

8.1 Calibration

Weigh to the nearest 0,1 mg approximately 40 mg of each solvent standard (5.1, 5.2 and respectively 5.3) each into a 10 ml volumetric flask and make up to volume with butan-1-ol (5.4) (calibration solutions A1, B1 and respectively C1).

Weigh to the nearest 0,1 mg approximately 20 mg of each solvent standard (5.1, 5.2 and respectively 5.3) each into a 10 ml volumetric flask and make up to volume with butan-1-ol (5.4) (calibration solutions A2, B2 and respectively C2).

Pipette 1ml of each calibration solution A1, B1 respectively C1 each into a 5 ml volumetric flask and make up to volume with butan-1-ol (5.4) (calibration solutions A3, B3 and respectively C3). Calculate the concentration of each standard solution in milligrams per 10 ml.

Pipette 1ml of each calibration solution A2, B2 respectively C2 each into a 5 ml volumetric flask and make up to volume with butan-1-ol (5.4) (calibration solution A4, B4 respectively C4). Calculate the concentration of each standard solution in milligrams per 10 ml.

Transfer each solution into an autosampler vial and inject 1 µl of each into the gas chromatograph. At least the quality of separation shown in Figure A.1 shall be achieved.

NOTE Manual injection is also suitable.

Record the peak areas and calculate the calibration curve for each solvent, using linear regression (least square) and without forced origin.

8.2 Determination

Weigh to the nearest 0,1 mg approximately 400 mg of the liquid detergent into a 10 ml volumetric flask and make up to volume with butan-1-ol (5.4).

Treat the solution in an ultrasonic bath (6.4) if necessary and filter through a membrane filter (6.5) if undissolved particles remain in the solution.

Fill an autosampler vial with the sample solution and inject 1 µl into the gas chromatograph. At least the quality of separation shown in Figure A.1 shall be achieved.

NOTE 1 Manual injection is also suitable.

Record the peak areas of the peaks of interest. Peak assignment is made by comparison of the retention times of the sample peaks with the retention times of the reference peaks.

NOTE 2 In cases where peak assignment is not clear, the solvent of interest should be added to the sample solution and then the gas chromatography should be repeated.

9 Calculation and expression of results

The content of the low boiling solvent i, w_i, expressed in grams per 100 g is calculated according to the following equation:

$$w_i = \frac{A_i - b_i}{a_i} \times \frac{100}{m} \quad (1)$$

where

A_i is the peak area of the low boiling solvent i in the sample in counts;

b_i is the axis intercept of the calibration curve of the solvent i in counts;

a_i is the slope of the calibration curve of solvent i in counts/milligrams per10 ml;

m is the mass of the sample, in milligrams.

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 and 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.

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.

Precision data are given in Annex B.

11 Test report

The test report shall include the following information:

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

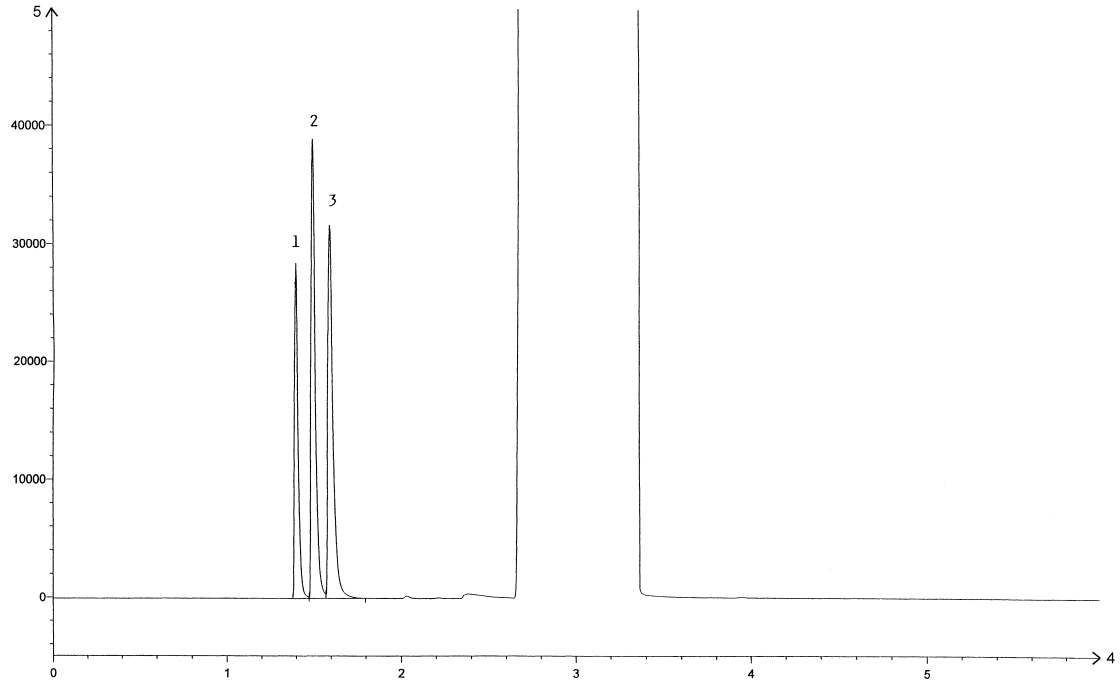
b) method used (a reference to this document);

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	Methanol	4	Time, in minutes
2	Ethanol	5	Response, in millivolts
3	Propan-2-ol		

Figure A.1 — Typical gas chromatogram

The following GC conditions have been found to be suitable:

- Column Head-pressure: 115 kPa;
- Split ratio: 1:50;
- Injection temperature: 200 °C;
- Temperature programme: 30 °C for 4 min, then to 120 °C at 10 °C/min, then to 280 °C at 40 °C/min
- Detector temperature: 280 °C;
- Injection volume : 1 µl.

Annex B
(informative)

Results of inter-laboratory test

The inter-laboratory test was carried out in 2002 by CESIO/AISE WG "Surfactant Analysis". Test sample 1 was a commercial softener formulation. Test sample 2 was a commercial dish washing liquid. The analyses were done using either automatic or manual injection into the gas chromatograph. The results of the inter-laboratory test were evaluated in accordance with ISO 5725-2.

Table B1 — Results of inter-laboratory test

Designation	**Propan-2-ol determination (sample 1)**	**Ethanol determination (sample 2)**
Number of laboratories participating	12	12
Number of laboratories not eliminated	10	10
Number of individual test results of all laboratories	36	36
Mean value, m, in g/100 g	1,17	3,09
Repeatability standard deviation, s_r, in g/100 g	0,025	0,074
Repeatability limit, r ($s_r \times 2{,}8$), in g/100 g	0,07	0,21
Variation coefficient of repeatabilityin %	2,16	2,40
Reproducibility standard deviation, s_R, in g/100 g	0,091	0,132
Reproducibility limit, R ($s_R \times 2{,}8$), in g/100 g	0,25	0,37
Variation coefficient of reproducibility, in %	7,75	4,27
NOTE Two laboratories were eliminated, because they did not follow the calibration procedure as it is described in the method.		

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