BRITISH STANDARD

BS EN
14666:2005

Surface active Agents — Determination of tetraacetyl ethylene diamine (TAED) content in TAED granules — Gas chromatographic method

The European Standard EN 14666:2005 has the status of a British Standard

ICS 71.100.40

National foreword

This British Standard is the official English language version of EN 14666:2005.

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 international/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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Summary of pages

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EN 14666

June 2005

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English version

Surface active Agents - Determination of tetraacetyl ethylene diamine (TAED) content in TAED granules - Gas chromatographic method

Agents de surface - Détermination de la teneur en tétra-acétyl-éthylène diamine (TAED) dans les granulés de TAED - Méthode par chromatographie en phase gazeuse

Grenzflächenaktive Stoffe - Bestimmung des Tetraacetylethylendiamin (TAED)-Gehalts in TAED-Granulaten - Gaschromatographisches Verfahren

This European Standard was approved by CEN on 19 May 2005.

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Contents

Foreword

This document (EN 14666:2005) 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of an identical text or by endorsement, at the latest by December 2005, and conflicting national standards shall be withdrawn at the latest by December 2005.

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1 Scope

This European Standard specifies a method for the determination of tetraacetyl ethylene diamine (TAED) content greater than or equal to 25 g/100 g in granules.

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 Principle

The TAED granules are dissolved in acetonitrile, the internal standard is added to this solution and finally it is injected to a gas chromatograph. A flame ionisation detector is used for detection. The results are calculated from the peak areas taking into account the response factors that are determined previously.

4 Reagents

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

4.1 Acetonitrile, C_2H_3N.

4.2 Stearic acid methyl ester, $C_{19}H_{38}O_2$, ≥ 99 % (m/m).

4.3 TAED, $C_{10}H_{16}O_4N_2$, ≥ 99 % (m/m).

4.4 Internal standard solution

Weigh into a 100 ml volumetric flask 250 mg of the stearic acid methyl ester (4.2) to the nearest 0,1 mg and make up to the mark with acetonitrile (4.1).

4.5 Carrier gas, for gas chromatography.

4.6 Auxiliary gases, for gas chromatography.

5 Apparatus

Normal laboratory apparatus and the following:

5.1 Gas chromatograph, with split/splitless injector, flame ionisation detector and integrator or computer.

5.2 Fused silica capillary column, capable of the separation characteristics shown in Figure A1.

NOTE A 25 m length, 0,25 mm internal diameter and 0,25 μm film thickness fused silica capillary column with 100% crosslinked dimethyl-polisiloxane stationary phase is advisable.

5.3 **Syringe**, capacity 5 µl.

5.4 **Filter**, porosity 0,45 µm.

6 Sampling and preparation of the sample

The laboratory sample shall be prepared and stored in accordance with ISO 607. Granules shall be ground for 1 min in an analytic mill or in a mortar to reach homogeneity.

7 Procedure

7.1 Calibration

Weigh into sample vials 25 mg, 50 mg, 75 mg and 100 mg of TAED (4.3) to the nearest 0,1 mg.

NOTE It is not required to perform the calibration before each series of samples. However a standard of 50 mg of TAED in 20 ml of the internal standard solution (4.4) should be analysed before each measurement. The value of response factor should not differ by more than 0,5 % from the average value of the calibration.

Add 20 ml of the internal standard solution (4.4) and dissolve the samples.

Inject each calibration solution three times into the gas chromatograph (5.1). An example of a calibration chromatogram is given in Figure A.1.

Record the peak areas of TAED and internal standard for each chromatogram and carry out a linear regression analysis of (A_{IS}/A_I) versus (m_{IS}/m_I). The response factor of TAED referring to the internal standard is the slope of the calculated calibration curve.

If a correlation factor of less than 0,98 is obtained prepare new calibration solution and re-inject.

If a data acquisition system is not available, calculate the response factor RF_i for each chromatogram according to the following formula:

$$RF_i = \frac{A_{IS} \times m_I}{A_I \times m_{IS}} \quad (1)$$

where

A_I the peak area of TAED in the chromatogram i of calibration solution;

A_{IS} the peak area of the stearic acid methyl ester (4.2) in the chromatogram i of calibration solution;

m_I the mass of TAED in the calibration solution of the chromatogram i, in milligrams;

m_{IS} the mass of the stearic acid methyl ester in the calibration solution of the chromatogram i, in milligrams.

The response factor RF of TAED referring to the internal standard is the mean value of all RF_i. If the relative standard deviation of RF is higher than 3 % repeat the calibration procedure and re inject.

7.2 Determination

Immediately after grinding or crushing the sample (see Clause 6), weigh approximately 500 mg of the sample (*m*) to the nearest 0,1 mg into a 100 ml volumetric flask. Add about 250 mg stearic acid methyl ester (4.2) to the nearest 0,1 mg and make up to the mark with acetonitrile (4.1).

Insert a stirring rod into the flask and stir for 20 min on a magnetic stirrer at room temperature.

Inject the clear solution directly into the gas chromatograph (5.1). If the solution is cloudy, filter it through the filter (5.4) and only then inject it into the gas chromatograph.

Set up the gas chromatograph to give results similar to the example chromatogram in Figure A.1.

8 Calculation and expression of results

The content of TAED, $w(C_{10}H_{16}O_4N_2)$, in the sample, expressed in grams per 100 g, is calculated by the equation (2):

$$w(C_{10}H_{16}O_4N_2) = \frac{RF \times m_{IS} \times A_S \times 100}{m \times A_{IS}} \quad (2)$$

where

RF the response factor of TAED relating to the stearic acid methyl ester (4.2), (see 7.1);

m_{IS} the mass of the stearic acid methyl ester, in milligrams (see 7.2);

m the mass of the sample, in milligrams (see 7.2);

A_{IS} the peak area of the stearic acid methyl ester in the chromatogram of the sample solution;

A_S the peak area of TAED in the chromatogram of the sample solution.

9 Precision

9.1 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.

Precision data are given in Annex B.

9.2 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 the 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;

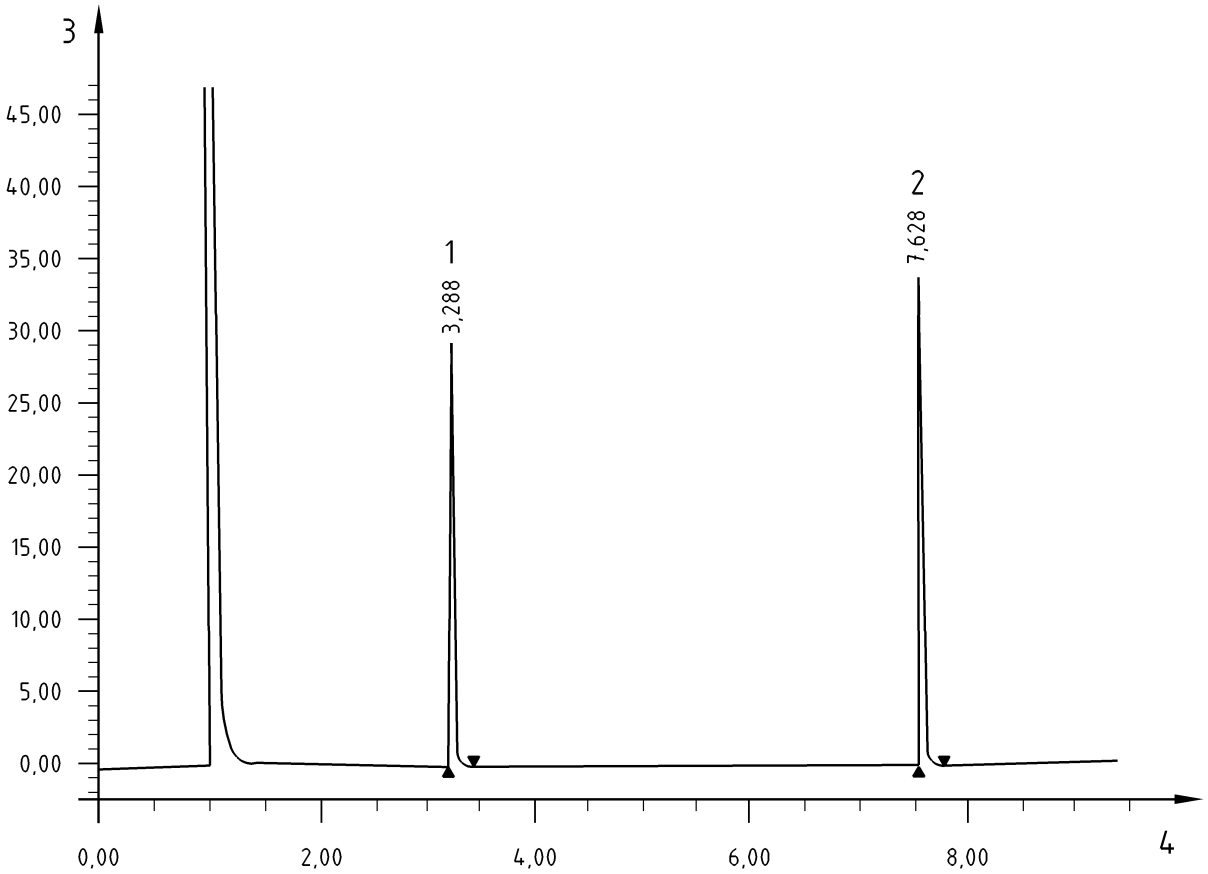
b) method used (a reference to this document);

c) test results;

d) details of any operation 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 like to have affected the results.

Annex A
(informative)

Typical gas chromatogram



Key

1 TAED
2 stearic acid methyl ester (internal standard)
3 voltage, in millivolts
4 time, in minutes

Figure A1 — Gas chromatogram of a calibration solution with 50 mg of TAED

An example of a suitable set of conditions is:

- Carrier gas: hydrogen or helium;
- Column head pressure: 150 kPa (1,5 bar);
- Split: 50 ml/min;
- Injection volume: 1,5 µl;
- Injector temperature: 250 °C;
- Detector temperature: 300 °C;
- Temperature programme: 150 °C to 220 °C with 10 °C/min;
  220°C to 300°C with 15 °C/min.

Annex B
(informative)

Results of inter-laboratory test

The inter-laboratory test was carried out in 1999 by the German “Common Committee for Analysis of Surfactants” (GAT). The test sample was a commercial TAED granule. The results of the inter-laboratory test were evaluated in accordance with ISO 5725-2.

Table B1 — Results of inter-laboratory test

Designation	Precision data
Numbers of laboratories participating	6
Numbers of laboratories not eliminated	6
Number of individual test results of all laboratories	18
Mean value, m, in g/100 g	86,19
Repeatability standard deviation s_r, in g/100 g	0,304
Repeatability limit r (s_r x 2,8), in g/100 g	0,850
Reproducibility standard deviation s_R, in g/100 g	0,452
Reproducibility limit R (s_R x 2,8), in g/100 g	1,266
Repeatability coefficient of variation, in %	0,352
Reproducibility coefficient of variation, in %	0,524

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