BRITISH STANDARD

BS EN
12836:2002

Surface active agents — Determination of the water number of alkoxylated products

The European Standard EN 12836:2002 has the status of a British Standard

ICS 71.100.40

National foreword

This British Standard is the official English language version of EN 12836:2002. It supersedes DD ENV 12836:2001 which is withdrawn.

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.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled "International Standards Correspondence Index", or by using the "Find" facility of the BSI Standards Electronic Catalogue.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

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 11 April 2002

Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 9 and a back cover.

The BSI copyright date displayed in this document indicates when the document was last issued.

Amendments issued since publication

Amd. No.	Date	Comments

ISBN 0 580 39491 3

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 12836

March 2002

ICS 71.100.40

Supersedes ENV 12836:1999

English version

Surface active agents - Determination of the water number of alkoxylated products

Agents de surface - Détermination du nombre d'eau dans les produits alkoxylés

Grenzflächenaktive Stoffe - Bestimmung der Wasserzahl von alkoxylierten Produkten

This European Standard was approved by CEN on 3 January 2002.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 12836:2002 E

Contents

Foreword

This document EN 12836:2002 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 September 2002, and conflicting national standards shall be withdrawn at the latest by September 2002.

This document supersedes ENV 12836:1999.

Significant technical differences between this standard and ENV 12836:1999 are the deletion of annexes B and C and the modification of annex A relating of precision data.

Annex A is informative.

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, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

H.L. Greenwald, G.L. Brown and M.N. Fineman [1] and W.C. Griffin [2] proposed the so-called water number as an easy-to-measure criterion for the hydrophilic-lipophilic-balance (HLB) value of surface active agents and oils. The method described was based on a dioxane/benzene mixture. In the following test method noxious benzene has been replaced by toluene. The water number is not altered by this change.

It is also possible to replace dioxane with triethylene glycol dimethyl ether. This, however, results in a change in the absolute numerical value of the water number, especially for high molecular mass nonionics (for example ethylene oxide/propylene oxide (EO/PO) block polymers).

1 Scope

This European Standard specifies the determination of the water number of ethoxylated products up to about 70 % ethylene oxide. EO/PO block polymers with water numbers higher than about 23 become hard to interpret.

NOTE The values obtained by this method are not completely identical with the water number obtained in dioxane, in particular water numbers higher than 23.

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).*

EN ISO 4320, *Non-ionic surface active agents - Determination of cloud point index - Volumetric method (ISO 4320:1977).*

3 Term and definition

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

3.1
water number
number of millilitres of water required to bring about a persistent turbidity in a solution with a specified volume of a solvent mixture at a constant temperature

NOTE Results of water numbers are very sensitive to temperature deviations.

4 Principle

To a solvent mixture consisting of dioxane/toluene (or triethylene glycol dimethyl ether/toluene) in which the sample is dissolved to give a clear solution, water is added at a temperature of (25 ± 1) °C until a persistent turbidity appears.

5 Reagents

All reagents shall be of a recognized analytical grade and the water used shall conform to grade 3, in accordance with EN ISO 3696.

5.1 Solvent mixture, with a mass fraction of 97,0 % dioxane and 3,0 % toluene.

NOTE If dioxane is not wanted or cannot be used, it may be replaced by triethylene glycol dimethyl ether (purity ≥ 98 %).

6 Apparatus

Ordinary laboratory apparatus and the following :

6.1 100 ml conical flask with glass stopper.

6.2 2 x 50 ml semi-microburettes or thermostatable (double-walled) titration equipment.

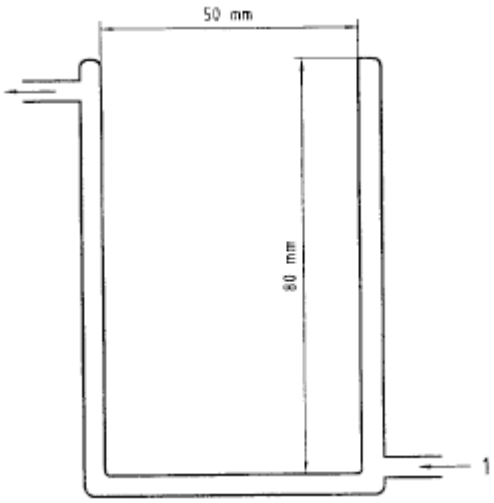
6.3 Double-walled beaker (see Figure 1).

6.4 Thermostat with circulating pump, adjustable to ± 0,1 °C.

6.5 Analytical balance, accurate to ± 0,01 g.

6.6 Magnetic stirrer.

Dimensions in millimetres



Key

1 Direction of water circulation

Figure 1 — Double-walled beaker in accordance with EN ISO 4320

7 Preparation of test sample

For the determination of the water number, 100 % active substance shall be used. If the sample contains solvent, it shall be completely removed (by distillation for example rotating evaporation).

8 Procedure

8.1 Dissolution of the sample

Weigh (1,00 ± 0,01) g of substance (sample) into a dry flask (6.1) with ground glass joint or directly into the double-walled beaker (6.3). Into this flask or beaker pour 30,0 ml of solvent mixture (5.1) from a 50 ml semi-microburette (6.2) whose storage bottle stands in a thermostated bath at (25 ± 1) °C. When using a conical flask with a ground glass joint close it with a glass stopper and dissolve the sample, if necessary by heating slightly and briefly. The temperature to stabilize at (25 ± 1) °C.

8.2 Determination of water number

Pour the clear solution into the double-walled beaker (6.3), maintained at (25 ± 1) °C, stir with magnetic stirrer (6.6), covered with a polyethylene cover and drop in water at a rate of 1 ml/min to 2 ml/min from a second semi-microburette (6.2), whose storage bottle also stands in a thermostated bath at (25 ± 1) °C, until turbidity persists for 2 min.

9 Expression of results

Report the result as the mean of two titrations calculated to the nearest 0,1 ml.

10 Precision

10.1 Repeatability

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

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 method and the solvant used (dioxane or triethylene glycol dimethyl ether) ;

d) the test results ;

e) the test date ;

f) details of any operations not specified in this European Standard and any operations regarded as optional, as well as any incidents which may have influenced the results.

Annex A
(informative)

Interlaboratory test results

These precision data were obtained by a ring test carried out on five samples. The evaluation was performed in accordance with ISO 5725-2:

Table A.1 — Samples

Sample	Description
A	Ethoxylated nonylphenol 2 EO
B	Ethoxylated nonylphenol 4 EO
C	Ethoxylated nonylphenol 6 EO
D	Ethoxylated nonylphenol 8 EO
E	Ethoxylated nonylphenol 10 EO

Table A.2 — Precision data

Designation	Sample A	Sample B	Sample C	Sample D	Sample E
Number of participating laboratories	4	4	4	4	4
Number of laboratories not eliminated	4	4	4	4	4
Number of individual measurement of all laboratories	9	8	10	8	9
Mean value (ml)	17,08	19,77	21,81	23,40	24,35
Repeatability standard deviation s_r (ml)	0,06	0,06	0,06	0,06	0,06
Repeatability limit, r, (r = 2,8 x s_r) (ml)	0,17	0,17	0,17	0,17	0,17
Repeatability coefficient of variation (%)	1,00	0,86	0,78	0,73	0,70
Reproducibility standard deviation s_R (ml)	0,44	0,46	0,44	0,75	0,55
Reproducibility limit, R, (R = 2,8 x s_R) (ml)	1,23	1,28	1,23	2,10	1,54
Reproducibility coefficient of variation (%)	7,20	6,47	5,64	8,97	6,32

Bibliography

[1] H.L. Greenwald ; G.L. Brown et M.N Fineman : Analyt. Chemistry 28 (1956), 1693.

[2] W.C Griffin : Journ. Soc. Cosm. Chem. 1 (1949), 311.

[3] 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.*

BSI — British Standards Institution

BSI is the independent national body responsible for preparing British Standards. It presents the UK view on standards in Europe and at the international level. It is incorporated by Royal Charter.

Revisions

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions.

It is the constant aim of BSI to improve the quality of our products and services. We would be grateful if anyone finding an inaccuracy or ambiguity while using this British Standard would inform the Secretary of the technical committee responsible, the identity of which can be found on the inside front cover. Tel: +44 (0)20 8996 9000. Fax: +44 (0)20 8996 7400.

BSI offers members an individual updating service called PLUS which ensures that subscribers automatically receive the latest editions of standards.

Buying standards

Orders for all BSI, international and foreign standards publications should be addressed to Customer Services. Tel: +44 (0)20 8996 9001. Fax: +44 (0)20 8996 7001. Email: orders@bsi-global.com. Standards are also available from the BSI website at http://www.bsi-global.com.

In response to orders for international standards, it is BSI policy to supply the BSI implementation of those that have been published as British Standards, unless otherwise requested.

Information on standards

BSI provides a wide range of information on national, European and international standards through its Library and its Technical Help to Exporters Service. Various BSI electronic information services are also available which give details on all its products and services. Contact the Information Centre. Tel: +44 (0)20 8996 7111. Fax: +44 (0)20 8996 7048. Email: info@bsi-global.com.

Subscribing members of BSI are kept up to date with standards developments and receive substantial discounts on the purchase price of standards. For details of these and other benefits contact Membership Administration. Tel: +44 (0)20 8996 7002. Fax: +44 (0)20 8996 7001. Email: membership@bsi-global.com.

Information regarding online access to British Standards via British Standards Online can be found at http://www.bsi-global.com/bsonline.

Further information about BSI is available on the BSI website at http://www.bsi-global.com.

Copyright

BSI
389 Chiswick High Road
London
W4 4AL