

SLOVENSKI STANDARD SIST ENV 12836:2001

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

Grenzflächenaktive Stoffe - Bestimmung der Wasserzahl von alkoxylierten Produkten

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

Ta slovenski standard je istoveten z: (standards iteh ai) ENV 12836:1999

<u>SIST ENV 12836:2001</u>

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ICS:

71.100.40 Površinsko aktivna sredstva Surface active agents

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EUROPEAN PRESTANDARD PRÉNORME EUROPÉENNE EUROPÄISCHE VORNORM

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ICS 71.100.40

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 Prestandard (ENV) was approved by CEN on 9 October 1999 as a prospective standard for provisional application.

The period of validity of this ENV is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the ENV can be converted into a European Standard.

CEN members are required to announce the existence of this ENV in the same way as for an EN and to make the ENV available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the ENV) until the final decision about the possible conversion of the ENV into an EN is reached.

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

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

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Foreword

This European Prestandard has been prepared by Technical Committee CEN/TC 276 "Surface active agents", the secretariat of which is held by AFNOR.

Annexes A, B, and C are for information.

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

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Introduction

H.L. Greenwald, G.L. Brown and M.N. Finemann [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).

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1 Scope

This European Prestandard specifies the determination of the water number of ethoxylated products with 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 Prestandard 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 Prestandard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

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 Terms and definitions eh STANDARD PREVIEW

For the purposes of this European Prestandard, the following definition applies:

3.1

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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 number determination 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 In case dioxane is not wanted or cannot be used, it may be replaced by triethylene glycol dimethyl ether (purity \geq 98 %).

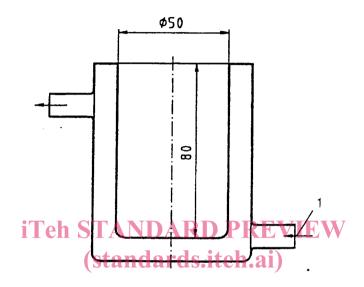
6 Apparatus

- **6.1 100 ml conical flask** with glass stopper.
- 6.2 2 x 50 ml semi-microburettes or thermostatable (double-walled) titration equipment.

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- 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 millimeters



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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 $(1,00\pm0,01)$ 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 persistes for 2 min.

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 repeatability conditions are conditions where mutually independent test results are obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within short intervals of time.

Ring test results are shown in annexes A and B.

10.2 Reproducibility

The reproducibility conditions are conditions where test results are obtained with the same method on identical test material in different laboratories with different operators using different equipment.

Ring test results are shown in annexes A and C.

11 Test report

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The test report shall include the following information:

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all information necessary for the complete identification of the sample;

a reference to this European Prestandard atalog/standards/sist/19eba6a3-0cf5-4e7d-ab93b)

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- the method and the solvant used (dioxane or triethylene glycol dimethyl ether);
- the test results; d)
- the test date;
- details of any operations not specified in this European Prestandard and any operations regarded as optional, f) as well as any incidents which may have influenced the results.