SLOVENSKI STANDARD
SIST EN 1890:1999

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Površinsko aktivne snovi - Določanje točke zmotnitve neionskih površinsko aktivnih snovi, pridobljenih s kondenzacijo etilen oksida

Surface active agents - Determination of cloud point of non-ionic surface active agents obtained by condensation of ethylene oxide

Grenzflächenaktive Stoffe - Bestimmung des Trübungspunktes nichtionischer, durch Anlagerung von Ethylenoxid hergestellter grenzflächenaktiver Stoffe

Agents de surface - Détermination du point de trouble des agents de surface non ioniques obtenus par condensation d'oxydes d'éthylene

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ICS:

71.100.40 Površinsko aktivna sredstva Surface active agents

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English version

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This European Standard was approved by CEN on 13 February 1999.

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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 Central Secretariat has the same status as the official versions.

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

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Contents

Foreword

This European Standard 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of an identical text or by endorsement, at the latest by September 1999, and conflicting national standards shall be withdrawn at the latest by September 1999.

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

Introduction

Solutions of non-ionic surface active agents obtained by the reaction of ethylene oxide with a hydrophobic base molecule, in water or in mixtures of water and organic solvents become turbid at a given temperature as the temperature increases and finally separate into two liquid phases. The process is reversible and the system becomes homogeneous again upon cooling. The temperature at which the solution becomes clear upon cooling is determined as the "cloud point". This temperature is characteristic for a particular surfactant. This temperature increases with the amount of ethylene oxide chemically combined in the surfactant molecule for a given composition of solvents.

This phenomenon is not limited to ethoxylated surfactants and the cloud point can be determined also for other non-ionic compounds.

The knowledge of the cloud point of non-ionic surfactants obtained by the reaction of ethylene oxide with hydrophobic bases is important for their use. For a given base molecule, the cloud point is indeed a simple measure of the amount of the combined ethylene oxide. Moreover, the cloud point suggests directly the temperature at which many functional surfactant properties change dramatically. The curve of cloud point versus degree of ethoxylation is asymptotic, therefore molecules containing high amounts of ethylene oxide show only small differences in their cloud point. In these cases the cloud point loses its significance.

Methods D and E of this European Standard are based on methods described in ISO 1065 and DIN 53917 respectively. The test principle is the same and test conditions are similar but more exactly defined.

1 Scope

This European Standard specifies methods for the determination of the cloud point of solutions of non-ionic surface active agents obtained by the reaction of ethylene oxide with a hydrophobic base molecule.

This standard primarily applies to surfactants obtained by reaction of ethylene oxide with hydrophobic base molecules, such as fatty alcohols, fatty acids, long-chain alkylphenols, fatty amines, fatty acid esters of sugar derivatives among other ethoxylated non-ionic surfactants, which are by far the most commonly used.

NOTE Other non-ionic surfactants containing other structural units, such as propylene oxide-ethylene oxide block copolymers, have distinctive behaviours that make the determination of the cloud point more difficult. This leads sometimes to a continuous turbidity over a temperature range of several degrees or even to the occurrence of two cloud points at significantly different temperatures.

2 Normatives 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.

EN ISO 862, *Surface active agents – Vocabulary (ISO 862:1984 + Corrigendum 1:1993).*

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987).*

ISO 607:1980, *Surface active agents and detergents - Methods of sample division.*

3 Terms and Definitions

For the purposes of this Standard, the definitions given in EN ISO 862 and the following apply :

3.1
cloud temperature
temperature above which aqueous solutions of certain non-ionic surface active agents become heterogeneous by the separation into two liquid phases (coacervation) [EN ISO 862]

NOTE 1 The value of the cloud temperature depends on the concentration of the solution.

NOTE 2 The temperature at which the system becomes homogeneous upon cooling is called "temperature of clarification". The cloud temperature and the temperature of clarification do not need to coincide for reasons concerned with the measurement procedure. However, for practical reasons, the temperature of clarification is conventionally called the "cloud point".

3.2
cloud point
critical lower phase-separation temperature (lower consolute temperature) above which the system is a cloudy solution and a further temperature rise results in two immiscible phases that are in equilibrium

NOTE 1 The cloud point is measured as the temperature falls.

NOTE 2 The cloud point depends on the number of ethylene oxide units linked to the base molecule and on their statistical distribution. It is also very sensitive to the presence of electrolytes and other organic substances in the aqueous solution. Therefore it should operate under well established conditions.

3.3
temperature of clarification
temperature at which the mixture of two liquid phases of aqueous solution of certain non-ionic surface active agents exhibiting a cloud temperature, becomes homogeneous on cooling [EN ISO 862]

NOTE The temperature of clarification is often determined as "cloud point".

4 Principle

The surfactant solution is heated until it is completely cloudy. Then it is slowly cooled with constant stirring while measuring the temperature. The temperature at which the turbidity disappears and the solution becomes homogeneous is recorded as the cloud point. Depending on the nature of the surfactant and the purity of the materials the solution can become completely clear or slightly opalescent but in this case a definite change from the cloudy solution is observed.

5 Reagents

During the test, unless otherwise stated, use only reagents of recognized analytical grade.

5.1 Water according to grade 3 in EN ISO 3696 or water of at least equivalent purity.

5.2 Diethylene glycol mono-n-butylether (special quality) C_4H_9-O-CH_2-CH_2-O-CH_2-CH_2-OH (also commercially known as butyldiglycol).

5.2.1 Specifications

The butyldiglycol (BDG) shall have the following :

- minimum purity : 99,5 % ;
- density at 20 °C : (0,952 ± 0,002) g/ml ;
- refractive index : n_{D20} = (1,431 ± 0,001) ;
- water content : < 0,1 %.

NOTE Impurities present in diethylene glycol mono-n-butylether and differences in concentration of its 25 % as mass fraction aqueous solution affect the cloud point to some extent. For arbitration purposes samples of diethylene glycol mono-n-butylether shall be exchanged between laboratories

5.2.2 Butyldiglycol/water, solution at mass fraction of 25 %

Dissolve 250 g of butyldiglycol (5.2.1) with 750 g of water.

5.3 Sodium chloride aqueous solutions

5.3.1 50,00 g NaCl per litre solution in water.

5.3.2 100,00 g NaCl per litre solution in water.

6 Apparatus

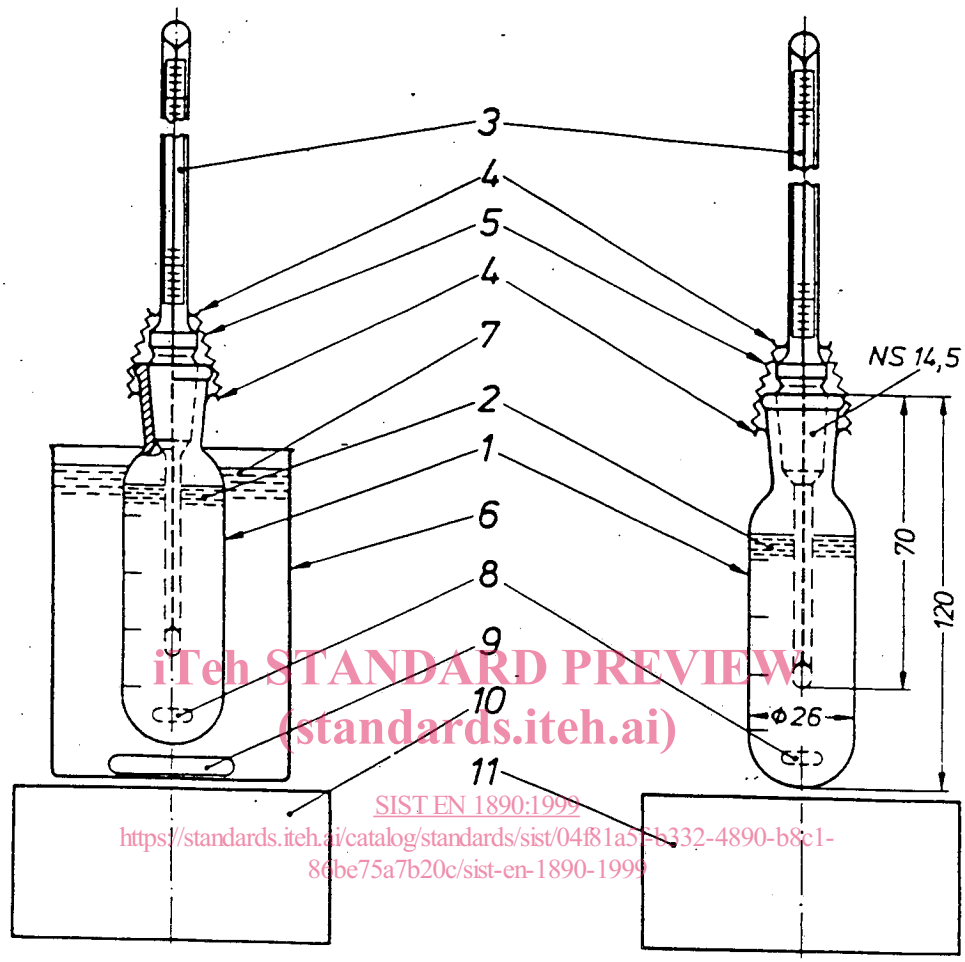
6.1 Calibrated thermometer, graduated in 0,1 °C, with a range appropriate to the temperature to be measured or a calibrated electronic measuring instrument with digital display.

6.2 Conical flask, capacity 250 ml, with glass or plastic stopper.

6.3 Test tube, capacity 30 ml.

6.4 Conventional heating appliance (see also Figure 1)

6.5 Beaker, capacity 250 ml, containing a transparent heat-transfer fluid.



Heating procedure

Cooling procedure

Key

1 Glass vessel to contain 25 ml of surfactant solution.
2 Surfactant solution.
3 Calibrated thermometer graduated in 0,1 °C, with NS 14,5 male ground glass point and immersion length of 90 mm. The range should be appropriate to the temperature to be measured. Calibrated electronic measuring instruments with digital display can also be used. Officially certified calibrated thermometers shall be used for arbitration purposes.
4 Aluminium hooks to attach the helical springs.
5 Helical springs (used in measurements above 90 °C).
6 600 ml beaker.
7 Heat transfer liquid (ethylene glycol).
8 Magnetic stirrer for thorough mixing of the surfactant solution.
9 Magnetic stirrer for thorough mixing of the heating liquid.
10 Heated magnetic stirrer.
11 Unheated magnetic stirrer.

Figure 1 - Example of a measurement system for determining the cloud point of non-ionic surfactants

7 Preparation and storage of samples

Prepare and store the sample of the surface active agent in accordance with ISO 607.