



# SLOVENSKI STANDARD

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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 grenzflächenaktive Stoffe

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

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English Version

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

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This European Standard was approved by CEN on 18 May 2006.

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 Central Secretariat 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 Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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## Foreword

This document (EN 1890:2006) 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of an identical text or by endorsement, at the latest by December 2006, and conflicting national standards shall be withdrawn at the latest by December 2006.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 1890:1999.

The main changes are:

- the purity of Butyldigycol was changed to correspond to commercially available products;
- the apparatus was simplified.

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, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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## 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.

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## 1 Scope

This document 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 document 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 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.

EN ISO 862:1995, *Surface active agents - Vocabulary - Trilingual version (ISO 862:1984/Cor 1:1993)*.

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

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

## 3 Terms and Definitions

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For the purposes of this document, the terms and definitions given in EN ISO 862:1995 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 862:1995]

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 862:1995]

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 :  $\geq 99$  % (m/m) (GC);
- density at 20 °C :  $(0,953 \pm 0,002)$  g/ml ;
- refractive index :  $n_D^{20} = (1,432 \pm 0,001)$ ;
- water content :  $\leq 0,1$  % (m/m).

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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 25 % (m/m).

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

Normal laboratory apparatus and the following:

**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 or other suitable vessel.

**6.3 Test tube**, capacity at least 30 ml, with a good temperature endurance.

**6.4 Conventional heating appliance**, for example Bunsen burner, hand-held hot air blower or thermostated oil bath.



## 7 Preparation and storage of samples

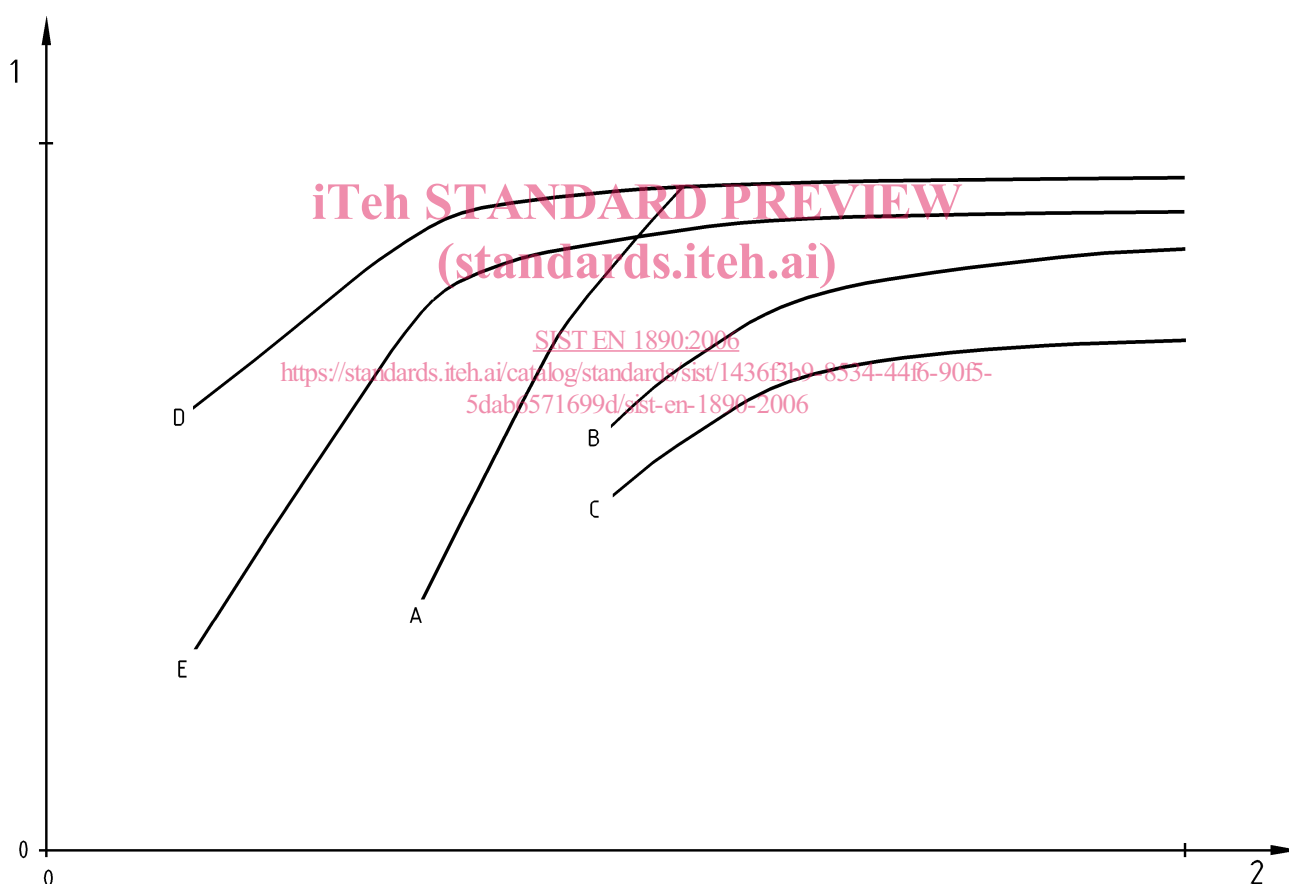
Prepare and store the sample of the surface active agent in accordance with ISO 607.

## 8 Procedure

### 8.1 Selection of test method

Five methods of cloud point determination are described; select from those given in 8.2 to 8.6, the most appropriate method for the particular surfactant under test, depending upon the temperature at which the solution of the test sample becomes turbid.

NOTE The temperature at which the solution of a surfactant becomes turbid depends on the ethylene oxide content (see Figure 1). The choice between methods A, B, C, D and E is based on the sharpness of the cloud point and the sensitivity of the method. It should be noted that the methods B and C and methods D and E will produce different cloud point results and so the method used should be fully noted when reporting any results.



#### Key

- 1 Temperature
- 2 Ethylene oxide content

- A 1 g sample + 100 g of water (5.1)
- B 1 g sample + 100 g of 50g/l NaCl solution (5.3.1)
- C 1 g sample + 100 g of 100g/l NaCl solution (5.3.2)
- D 5 g sample + 45 g of 25 % (m/m) BDG solution (5.2.2)
- E 5 g sample + 25 g of 25 % (m/m) BDG solution (5.2.2)

Figure 1 — Cloud point behaviour of typical fatty alcohol ethoxylates using different test methods