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

The European Standard EN 1890:2006 has the status of a British Standard

ICS 71.100.40

British Standards

National foreword

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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

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

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

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Contents

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of an identical text or by endorsement, at the latest by December 2006, and conflicting national standards shall be withdrawn at the latest by December 2006.

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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.

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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.

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

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 : ≥ 99 % (m/m) (GC);

— density at 20 °C : (0,953 ± 0,002) g/ml ;

— refractive index : n_D^{20} = (1,432 ± 0,001);

— water content : ≤ 0,1 % (m/m).

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

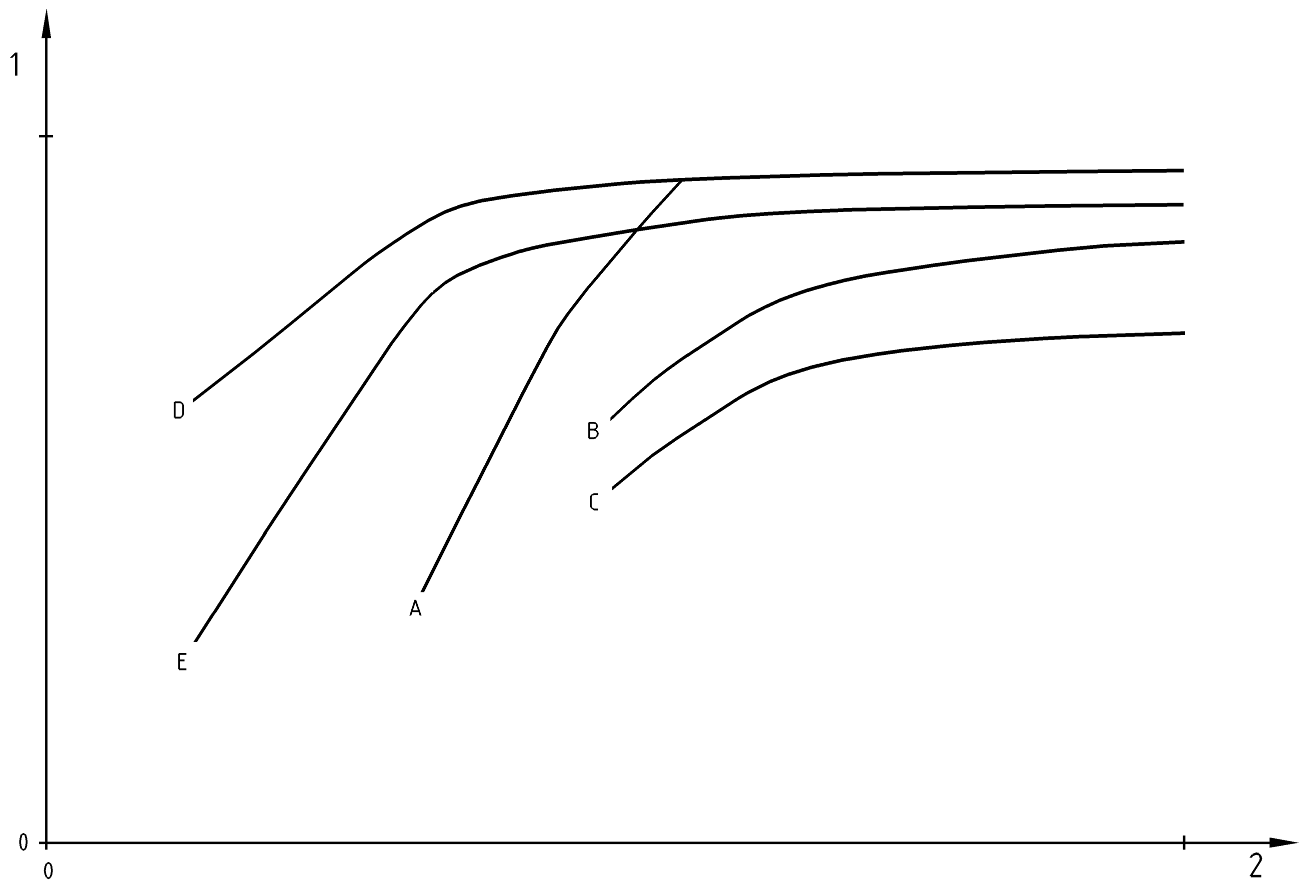
Prepare and store the sample of the surface active agent in accordance with ISO 607.

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

8.2 Method A : Water solution

NOTE 1 Method A is used when an aqueous solution of 1 g of sample plus 100 g of water becomes turbid at a temperature between 10 °C and 90 °C.

Weigh 1,0 g of the sample (see clause 7) to the nearest 0,01 g into the conical flask (6.2), add 100 g of water, stopper the flask and stir until the sample has dissolved to form a clear solution.

Pour about 15 ml to 25 ml of this solution into the test tube (6.3). Put the thermometer (6.1) into the tube and heat with a heating appliance (6.4) under slow stirring with the thermometer until it is completely opaque.

NOTE 2 The opacity appears as bands, which thicken, and results in turbidity. The temperature of the solution should not exceed by more than 10 °C the temperature at which the opacity appears.

Allow to cool slowly in the air while stirring. Read the temperature at which the opaque bands disappear.

Repeat the determination, using a fresh solution, until at least two results differ from each other by not more than 0,5 °C.

NOTE 3 A fresh solution is used to avoid loss of water due to evaporation.

8.3 Method B : Salt solution of NaCl at 50 g/l

NOTE Method B is used when an aqueous solution of 1 g of sample plus 100 g of water becomes turbid at a temperature above 90 °C.

Weigh 1,0 g of the sample (see clause 7) to the nearest 0,01 g into the conical flask (6.2), add 100 g of sodium chloride solution, c(NaCl) = 50 g/l (5.3.1) and stir until the sample has dissolved to form a clear solution.

Determine the cloud point using the procedure given in 8.2.

8.4 Method C : Salt solution of NaCl at 100 g/l

NOTE Method C is used when an aqueous solution of 1 g of sample plus 100 g of water becomes turbid at a temperature above 90 °C.

Weigh 1,0 g of the sample (see clause 7) to the nearest 0,01 g into the conical flask (6.2), add 100 g of sodium chloride solution, c(NaCl) = 100 g/l (5.3.2) and stir until the sample has dissolved to form a clear solution.

Determine the cloud point using the procedure given in 8.2.

8.5 Method D : Solution in 45 g of BDG/water (Butyldiglycol/water solution)

NOTE Method D is used when an aqueous solution of 1 g of sample plus 100 g of water becomes turbid at a temperature below 10 °C.

Weigh 5,0 g of the sample (see clause 7) to the nearest 0,01 g into the conical flask (6.2), add 45 g of a mass fraction of 25 % aqueous solution of diethylene glycol mono-n-butylether (5.2.2) and stir until the sample has dissolved to form a clear solution.

Determine the cloud point using the procedure given in 8.2.

8.6 Method E : Solution in 25 g of BDG/water (Butyldiglycol/water solution)

NOTE Method E is used when an aqueous solution of 1 g of sample plus 100 g of water becomes turbid at a temperature below 10 °C.

Weigh 5 g of the sample (see clause 7) to the nearest 0,01 g into the conical flask (6.2), add 25 g of a mass fraction of 25 % aqueous solution of diethylene glycol mono-n-butylether (5.2.2) and stir until the sample has dissolved to form a clear solution.

Determine the cloud point using the procedure given in 8.2.

9 Expression of results

Take as the result the arithmetic mean of at least two results that do not differ by more than 0,5 °C.

10 Precision

10.1 **Repeatability** (one observer, one apparatus):

If two results are determined under repeatable conditions by one observer, then both results are regarded as acceptable if they do not differ by more than 0,5 °C.

10.2 **Reproducibility** (different observers, different apparatus):

If a result is obtained under comparable conditions in each of two different laboratories, then both results are regarded as acceptable if they do not differ by more than 1,0 °C.

NOTE In measurements carried out for arbitration purposes and using diethylene glycol mono-n-butylether, the solvent samples should be also exchanged.

11 Test report

The test report shall include the following particulars:

a) all details required for complete identification of the sample;

b) method of determination by reference to EN 1890 and specific method (A to E) used;

c) cloud point in degrees to 0,1 °C ; individual values and mean value of at least two measurements, which results can differ by not more than 0,5 °C;

d) concentration of non-ionic surfactant in the solution if different from the recommended concentration;

e) any operation not included in this Standard, or regarded as optional;

f) date of the test.

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[1] H. Hoffmann, "Die Bestimmung des Trübungspunktes nichtionischer Tenside" Tenside Detergents 1(11), 30-31, (1974) (Part 6).

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[3] ISO 1065, *Non-ionic surface-active agents obtained from ethylene oxide and mixed non-ionic surface-active agents – Determination of cloud point.*

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