INTERNATIONAL STANDARD 1065

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Non-ionic surface active agents obtained from ethylene oxide — Determination of cloud temperature (cloud point)

Agents de surface non ioniques obtenus à partir de l'oxyde d'éthylène — Détermination de la température de trouble (point de trouble)

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 1065 was drawn up by Technical Committee ISO/TC 91, *Surface active agents*. It was submitted directly to the ISO Council, in accordance with clause 6.12.1 of the Directives for the technical work of ISO.

This International Standard cancels and replaces ISO Recommendation R 1065-1969, and Amendment 1-1972. ISO Recommendation R 1065 had been approved by the Member Bodies of the following countries :

Austria	Hungary	Portugal
Belgium	India	Romania
Canada	Iran	South Africa, Rep. of
Chile	Israel	Spain
Czechoslovakia	Japan	Sweden
Egypt, Arab Rep. of	Korea, Rep. of	Switzerland
France	Netherlands	Turkey
Germany	New Zealand	United Kingdom
Greece	Poland	Yugoslavia

No Member Body had disapproved the Recommendation.

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Non-ionic surface active agents obtained from ethylene oxide — Determination of cloud temperature (cloud point)

0 INTRODUCTION

Aqueous solutions of non-ionic surface active agents derived from ethylene oxide become heterogeneous when their temperature is raised, because of the formation of two liquid phases. The temperature above which this phenomenon occurs is the cloud temperature.

This phenomenon occurs at a temperature which is higher as the number of molecules of ethylene oxide combined in the product increases. The system becomes homogeneous when the temperature falls. The temperature at which the system becomes homogeneous is conventionally called the "temperature of clarification"[1].

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

The knowledge of the cloud temperature (cloud point) is of great importance in all applications, as the surface active properties vary very rapidly around this temperature.

The determination of the cloud temperature of a non-ionic product is a rapid and precise method of control. In manufacture, it is the quickest method of assessing the quantity of ethylene oxide attached to a given radical. However, it must be remembered that the curve relating cloud point and the extent of ethoxylation tends towards an asymptote; as a consequence, this method of checking is less precise for products with a long ethoxyl chain.

NOTE — The cloud temperature is a lower critical temperature of separation; above this critical temperature, the solution separates into two phases, and the appearance of these two phases when the temperature is raised leads to the clouding of the solution. This property is not specific to ethoxylated derivatives and it is possible to determine cloud temperatures for other types of derivatives.

This International Standard deals primarily with the determination of the cloud temperatures of non-ionic surface active agents derived from ethylene oxide by condensation on lipophilic base-molecules such as fatty alcohols, fatty amines, fatty acids, alkylphenols, esters of fatty acids and other molecules of the same type.

Other non-ionic surface agents, such as the ethylene oxide-propylene oxide block polymers, are known to have special properties which make it more difficult to determine their cloud temperatures.

Among these properties is the appearance of a progressive clouding over a temperature range of several degrees, and the existence of two very distinct cloud temperatures.

The cloud temperature is related to the number of ethylene oxide molecules fixed on the base-molecule, but it also depends on the concentration of the solution. It is therefore essential to operate the test at a clearly defined concentration.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies three methods for the measurement of the cloud temperature of non-ionic surface active agents derived from ethylene oxide by condensation on a lipophilic compound which has no oxypropylene groups.

The method chosen depends on the temperature at which the aqueous solution of the product being tested becomes cloudy.

2 REFERENCE

ISO 607, *Surface active agents — Methods of sample division.*[2]

3 SELECTION OF METHOD

NOTE — It may be impossible to measure the cloud temperature of certain very pure derivatives of ethylene oxide if dissolved in distilled water of very low conductivity. In such cases, the solution does not become heterogeneous at a definite temperature; instead, only a slight loss of clarity is observed.

However, replacement of the distilled water by an aqueous solution of sodium chloride (4 meq/l) enables the cloud temperature to be measured.

3.1 Method A

If the aqueous solution of the non-ionic surface active agent becomes cloudy at a temperature between 10 and 90 °C, carry out the measurement in distilled water (see 8.1).

1) See Addendum 1 to ISO/R 862, *Surface active agents — Glossary.*

2) In preparation. (Revision of ISO/R 607.)

3.2 Method B

If the aqueous solution of the non-ionic surface active agent becomes cloudy at a temperature lower than 10 °C, or if the product is not sufficiently soluble in water, carry out the measurement in an aqueous 25 % (*m/m*) solution of *n*-butyldiglycol (see 8.2). However, this method is not applicable to certain products with low ethylene oxide contents and which are insoluble in the aqueous 25 % (*m/m*) solution of *n*-butyldiglycol.

3.3 Method C

If the aqueous solution of the non-ionic surface active agent becomes cloudy at a temperature higher than 90 °C, carry out the measurement in a sealed tube (see 8.3), which makes it possible, by operating under pressure, to reach temperatures higher than the boiling point of the solution at atmospheric pressure.

It is also possible, if agreed in a contract, to determine the cloud temperature in salt solution, but this method is not so sensitive and the results cannot be related to the previous ones by a simple equation. This alternative method is indicated in the annex.

4 PRINCIPLE

Heating of the product in solution at the required concentration until the liquid is completely opaque. Cooling, with constant stirring. Observation of the temperature at which the opacity disappears.

5 REAGENT (for method B)

The water used for the different methods shall be distilled water or water of at least equivalent purity.

5.1 *n*-Butyldiglycol, 25 % (*m/m*) solution.

The *n*-butyldiglycol ($C_4H_9-O-CH_2-CH_2-O-CH_2-CH_2OH$) [2-(2-butoxyethoxy)-ethanol] shall have the following characteristics :

- density $\rho_{20} = 0{,}954 \pm 0{,}002$ g/ml;
- refractive index $n_D^{20} = 1{,}432 \pm 0{,}001$;
- water content less than 0,1 %.

NOTE – Even with the given specification, variable amounts of impurities in the *n*-butyldiglycol have a certain influence on the cloud temperature.

6 APPARATUS

Ordinary laboratory apparatus and

6.1 Conical flask, capacity 250 ml, complying with ISO/R 1773.

6.2 Thermometer, graduated in 0,1 °C, with a range appropriate to the temperature to be measured.

6.3 Graduated cylinder, capacity 100 ml.

6.4 Analytical balance.

6.5 Beaker, capacity 1 000 ml, containing transparent heat-transfer fluid (glycol, for example).

and, for Methods A and B :

6.6 Test tube, 20 mm in diameter and 150 mm long.

6.7 Conventional heating appliance.

for Method C (see figure) :

6.8 Ampoule, of safety glass covered with wire gauze, outside diameter 14 mm, inside diameter 12 mm, height 120 mm.

6.9 Magnetic stirrer with heater.

7 SAMPLING

The laboratory sample of surface active agent shall be prepared and stored according to the instructions given in ISO 607.

8 PROCEDURE

8.1 Method A (Use when cloudiness appears at a temperature between 10 and 90 °C)

8.1.1 *Test portion*

Weigh, to the nearest 0,01 g, exactly 0,5 g of the laboratory sample.

8.1.2 *Determination*

Place the test portion in the conical flask (6.1). Add 100 ml of distilled water, measured with the graduated cylinder (6.3). Stir until the test portion has dissolved or dispersed.

Place 15 to 20 ml of this solution in the test tube (6.6). Put the thermometer (6.2) in the tube, place the tube in the beaker (6.5) and heat, stirring the solution with the thermometer until it is completely opaque. (The opacity appears in the form of bands which thicken and then coalesce.) Allow to cool slowly while stirring. Read the temperature at which the opaque bands disappear on stirring; the liquid may stay clear or opalescent according to the nature of the non-ionic products and the purity of the raw materials used for their preparation. (See the note at the beginning of clause 3.)

Carry out several temperature measurements until at least two results differing by not more than 0,5 °C are obtained.

8.2 Method B (Use when cloudiness appears at a temperature below 10 °C, or the product is insoluble in water)

8.2.1 *Test portion*

Weigh, to the nearest 0,01 g, exactly 5 g of the laboratory sample.

8.2.2 *Determination*

Place the test portion in the conical flask (6.1). Add 45 g of the *n*-butyldiglycol solution (5.1). Stir until the test portion is dissolved.

Proceed as in method A (see 8.1.2).

8.3 Method C (Use when cloudiness appears at a temperature higher than 90 °C)

8.3.1 *Test portion*

Weigh, to the nearest 0,01 g, exactly 0,5 g of the laboratory sample.

8.3.2 *Determination*

Place the test portion in the conical flask (6.1). Add 100 ml of distilled water, measured with the graduated cylinder (6.3). Stir until the test portion has completely dissolved.

Transfer a quantity of this solution to the glass ampoule (6.8), to a depth of approximately 40 mm. Seal the ampoule by means of a flame and cover it with coarse wire gauze. Put it in the heat-transfer bath (6.5). The upper end of the ampoule should project slightly from the bath.

As a precaution against flying glass, place a protective screen of safety glass or transparent plastics in front of the apparatus since the ampoule may burst if it has not been well sealed. (See figure.)

Place the thermometer (6.2) in the heating fluid next to the sealed ampoule. Set the heater and the magnetic stirrer (6.9) in operation; stop the heating when the liquid contained in the ampoule becomes cloudy. Allow to cool while stirring the heat-transfer bath and note the temperature at which cloudiness disappears, as indicated for method A (see 8.1.2).

Carry out several temperature measurements until at least two results not differing by more than 0,5 °C are obtained.

9 EXPRESSION OF RESULTS

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

Indicate, to the first decimal place, the mean temperature at which the solution of the non-ionic surface active agent becomes either clear or opalescent again and the medium in which the measurement has been made.

Example :

Cloud temperature at 5 g/l in distilled water : . . .

Cloud temperature at 100 g/kg, in the presence of *n*-butyldiglycol : . . .

Cloud temperature at 5 g/l in sealed ampoule : . . .

9.1 Repeatability

The difference between the results of two determinations carried out simultaneously, or in rapid succession, on the same sample and by the same operator, should not exceed 0,5 °C.

9.2 Reproducibility

The difference between results obtained by two different laboratories on the same sample should not exceed 1 °C.

10 TEST REPORT

The test report shall include the following particulars :

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

b) the medium in which the measurement was carried out;

c) the reference of the method used;

d) the results and the method of expression used;

e) any unusual features noted during the determination;

f) any operation not included in this International Standard, or regarded as optional.

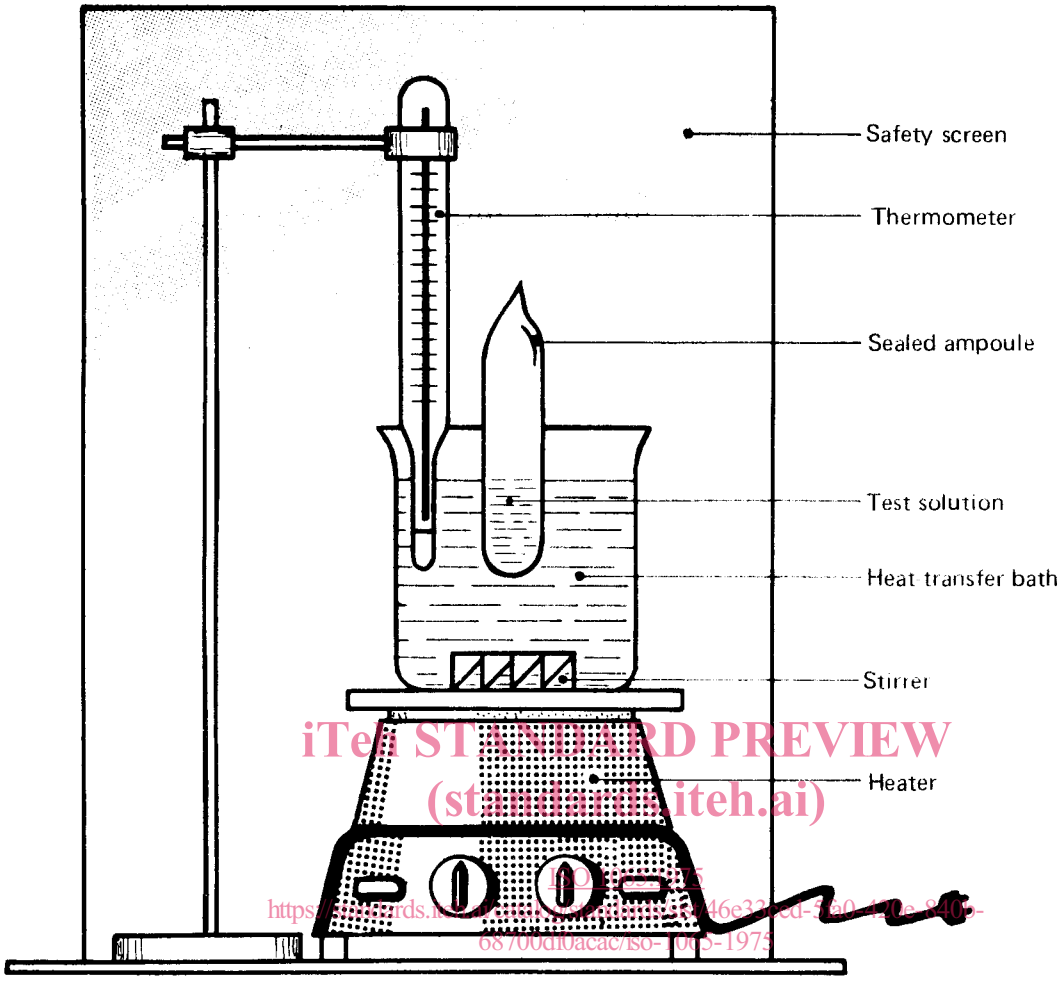

FIGURE — **Apparatus for method C**

ANNEX

CLOUD TEMPERATURE IN SALT SOLUTION

If agreed by contract, the cloud temperature can be determined in salt solution rather than in a sealed ampoule.

The technique is similar to that of method A (see 8.1), the surface active agent being dissolved in an aqueous 50 g/l solution of sodium chloride instead of in distilled water.