

# Surface active agents — Determination of pH value of solutions or dispersions

The European Standard EN 1262:2003 has the status of a  
British Standard

ICS 71.100.40

## National foreword

This British Standard is the official English language version of EN 1262:2003. It supersedes BS EN 1262:1996 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CII/34, Methods of test for surface active agents, which has the responsibility to:

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English version

## Surface active agents - Determination of pH value of solutions or dispersions

Agents de surface - Détermination de la valeur du pH des solutions ou des dispersions

Grenzflächenaktive Stoffe - Messung des pH-Wertes von Lösungen oder Dispersionen

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## Foreword

This document (EN 1262:2003) 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of an identical text or by endorsement, at the latest by April 2004, and conflicting national standards shall be withdrawn at the latest by April 2004.

This document supersedes EN 1262:1996.

Significant technical differences between this edition and EN 1262:1996 are as follows:

- a) addition of the specification of water and its preparation in 4.1;
- b) modification of the accuracy of the pH-meter in 5.1;
- c) introduction of the use of sodium chloride solution in 6.1 for solutions of low conductivity;
- d) introduction of a possibility in 6.2.1 for solution A to replace a mass fraction of 10% active matter solution;
- e) introduction of notes in 7.2 to indicate the possibility to avoid the absorption of CO<sub>2</sub> and to add few drops of diluted solution of sodium chloride for the measurement of the pH of non-ionic surface active agents.

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, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

## 1 Scope

This European Standard specifies a method for the determination of pH value of solutions or dispersions of surface active agents and gives a method for their preparation.

The method is applicable to aqueous solutions containing anionic, cationic and non-ionic surface active agents, ampholytic surface active agents and products in solution containing surface active agents.

## 2 Terms and definitions

For the purposes of this European Standard, the following term and definition apply.

### 2.1

#### **active matter**

all of the surface active agents responsible for a specified activity in a formulation

## 3 Principle

The pH value of the sample solution is determined by potentiometric measurement using a specific electrode and commercial pH meter.

## 4 Reagents

During the determination, unless otherwise specified, use only reagents of recognized analytical grade which have been checked in advance as to not interfere with the results.

**4.1 Water**, deionised water from which carbon dioxide has been removed and having a conductivity not exceeding 1 mS/m at 25 °C and a pH value between 6,0 and 7,0 or water of equivalent purity.

To remove carbon dioxide from the water it shall be boiled during not less than 10 min. Because water rapidly absorbs again CO<sub>2</sub>, it shall be allowed to cool down in a flask that has a tube filled with soda lime in its neck.

The pH value of the water shall be measured by addition of a few drops of dilute sodium chloride (4.4) in order to increase the conductivity.

**4.2 Standard buffer solutions**, two solutions which bracket the expected pH value of the sample solution, for adjusting the pH meter.

**4.3 Ethanol with a volume fraction of 96 %**, to determine its suitability, ethanol is mixed with deionised water (4.1), in a ratio of 50 : 50 (*m/m*) ; the pH value shall be between 6,0 and 7,0, otherwise a more suitable ethanol grade shall be used.

**4.4 Sodium chloride**, *w* (NaCl) = 10 % (aqueous solution).

## 5 Apparatus

Ordinary laboratory apparatus and the following.

**5.1 pH meter**, with a reading resolution of 0,01 pH, or better, comprising a potentiometric unit, a reference electrode and an electrode which is sensitive to hydrogen ions.

NOTE Combined electrodes of glass and calomel or Ag/Ag<sup>+</sup> are commercially available. They should be stored, cleaned and conditioned according to the manufacturer's instructions.

## 6 Preparation of product solutions or dispersions

### 6.1 General

Most surface active agents form clear dilute solutions in water.

Surface active agents which do not form a clear solution in water are first dissolved in ethanol and then mixed with water; this procedure sometimes produces dispersions which cream after a certain time, i.e. form two phases.

In case of non-ionic surface active agents, a few drops of sodium chloride (4.4) shall be added in order to increase the conductivity and hence the quality of the measurement.

The concentration of the solution to be tested has to be clearly specified. Because at very low surface active agent concentrations reading stability problems may arise, such situations are to be avoided. Three non-limitative situations are described in 6.2 and 6.3.

### 6.2 Water soluble products

#### 6.2.1 Solution A: mass fraction of 1 % active matter in water

Weigh, to the nearest 0,01 g, a test portion corresponding to 1 g active matter into a 150 ml beaker and make up to 100,00 g solution with water (4.1). If necessary, warm to not more than 50 °C to assist dissolution and cool to room temperature.

Many surface active agents require to use solutions with a mass fraction of 10 % active matter in water in order to obtain good reproducibility in which case replace solution A by a mass fraction of 10 % active matter solution, i.e. weigh a test portion corresponding to 10 g active matter into a 150 ml beaker and make up to 100,00 g solution with water (4.1).

#### 6.2.2 Solution B: mass fraction of 5 % solution in water

Weigh, to the nearest 0,01 g, a test portion of 5 g into a 150 ml beaker and make up to 100,00 g solution with water (4.1). If necessary, warm to not more than 50 °C to assist dissolution and cool to room temperature.

### 6.3 Sparingly water-soluble or water insoluble products

#### Solution C: mass fraction of 5 % sample in ethanol/water mixture

Weigh, to the nearest 0,01 g, a test portion of 5 g into a 150 ml beaker and add 47,5 g ethanol (4.3). If necessary, warm to not more than 50 °C to assist dissolution. Add 47,5 g water (4.1) and cool to room temperature.

NOTE For water-insoluble samples the addition of water produces a finely dispersed precipitate.

If required by a customer or product specification, the solvent or the quantity of test portion used to prepare solutions A, B and C may be different from those specified in 6.2 and 6.3. In this case the solvent and the concentration of the sample solution used shall be specified in the test report.

### 6.4 Highly concentrated formulations of surface active agents

Concerning measurements in highly concentrated solutions of surface active agents or molten products they can only be compared if all parameters and test conditions are defined.

At high concentrations the measurements obtained do not correspond to scientific pH definition.

## 7 Procedure

### 7.1 Adjustment of the pH meter

Adjust the pH meter (5.1) according to the manufacturer's instructions using the two standard buffer solutions (4.2).

### 7.2 Determination

Prepare two different solutions of the sample solution (A, B or C) to enable duplicate measurements to be carried out.

Wash the electrodes first with water (4.1) and then with a little of one portion of the sample solution. Immerse the electrodes in the first sample solution, stir slowly. If using a pH meter which has the facility, set the pH-meter to record the stabilised value.

NOTE 1 If the pH meter does not have this facility, the reading should be noted 10 s after a stable value has been achieved.

Remove the electrodes and rinse with a little of the second sample solution. Immerse the electrodes in the second sample solution and repeat the pH measurement.

If the two measurements differ by more than 0,1 pH unit, repeat the whole test, and if the situation persists, check the electrode and the pH meter.

Carry out all pH measurements at a temperature in the range from 20 °C to 25 °C.

If the sample is to be measured at a temperature not between 20 °C and 25 °C, the temperature knob of the instrument has to be reset at the sample temperature, and this circumstance has to be reported.

NOTE 2 In the case of cationic surface active agents, it is advisable to check the pH meter after each measurement, and readjust if necessary.

NOTE 3 When measuring the pH value the absorption of atmospheric carbon dioxide should be avoided.

NOTE 4 Addition of a few drops of a dilute solution of sodium chloride facilitates the measurement of the pH of non-ionic surface active agents.

## 8 Expression of results

Calculate the mean value of the two pH value readings to the nearest 0,1 pH unit at the test temperature.

## 9 Precision

### 9.1 Repeatability limit

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will not exceed the repeatability limit,  $r$ , in more than 5 % of cases.

Precision data are given in annex A.



## 9.2 Reproducibility limit

The absolute difference between two independent single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, will not exceed the reproducibility limit,  $R$ , in more than 5 % of cases.

Precision data are given in annex A.

If a reproducibility limit of less than 0,3 pH unit is aimed for, all relevant influencing parameters such as temperature compensation, electrode slope, calibration method, alkali error/acid error, dilution factor shall be very precisely described.

## 10 Test report

The test report shall include the following:

- a) all information necessary for the complete identification of the sample;
- b) the solvent composition used (if not water), and surface active agent concentration;
- c) the two standard buffer solutions used for the adjustment of the pH meter;
- d) the exact temperature of the measurements, with limit deviation of  $\pm 1$  °C;
- e) the age of the solution or dispersion at the time of conducting the measurements;
- f) the appearance of the solution or dispersion (clear, cloudy, creamy) at the time of conducting the measurements;
- g) the test results;
- h) any operation not included in this standard or regarded as optional, as well as any incidents which can have affected the results.

## Annex A (informative)

### Ring test results

The ring test was carried out from AEN/CTN 55B in 2001. The symbols used in table A.1 mean the following:

A: Alkylether sulfate, 70 % (active matter). Sample solutions of 2 % active matter.

B: Alkylether sulfate alkaline 26 % (active matter).

C: Alkylether sulfate alkaline 26 % (active matter). Sample solutions of 1 % active matter.

D: Fatty alcohol ethoxylated alkylchain C<sub>12</sub> C<sub>14</sub> with 4 EO. Sample solutions of 5 % active matter.

E: Cocamidopropylbetaine 30 % (active matter).

F: Cocamidopropylbetaine 30 % (active matter). Sample solutions of 1 % active matter.

G: Esterquat. Sample solution of 5 % active matter.

H: Alkylether sulfate alkaline 26 % (active matter).

Table A.1 — Ring test results of the pH determination

Designation	Sample A		Sample B		Sample C		Sample D		Sample E		Sample F		Sample G		Sample H	
	10	60	10	60	10	60	10	60	10	60	10	60	10	60	10	60
Reading time, s	10	60	10	60	10	60	10	60	10	60	10	60	10	60	10	60
Number of participating laboratories	11	11	11	11	11	11	11	11	11	11	11	11	11	11	11	11
Number of laboratories retained after eliminating outliers	11	11	11	11	11	11	11	11	11	11	11	11	11	11	11	11
Number of individual measurements of all laboratories	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2
Mean value, $m$	7,00	6,99	11,75	11,75	9,95	9,96	6,57	6,57	5,15	5,15	4,77	4,77	3,39	3,41	11,68	11,68
Repeatability standard deviation, $s_r$	0,018	0,019	0,017	0,015	0,026	0,025	0,018	0,021	0,019	0,016	0,012	0,010	0,030	0,030	0,019	0,018
Repeatability limit, $r$ ( $r=2,8 \times s_r$ )	0,050	0,053	0,048	0,043	0,074	0,069	0,051	0,058	0,054	0,044	0,034	0,027	0,084	0,085	0,052	0,050
Repeatability coefficient of variation, (%)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Reproducibility standard deviation, $S_R$	0,100	0,101	0,182	0,175	0,243	0,242	0,152	0,153	0,067	0,071	0,074	0,074	0,167	0,148	0,183	0,174
Reproducibility limit, $R$ ( $R=2,8 \times S_R$ )	0,280	0,282	0,510	0,490	0,682	0,677	0,425	0,428	0,188	0,198	0,208	0,208	0,468	0,415	0,513	0,488
Reproducibility coefficient of variation, (%)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
NOTE	The concept of variation coefficient is only relevant when the analytical response is directly related to the concentration of analyte present, which is not the case when pH is concerned.															
	Plots of the values $r$ and $R$ against $m$ do not indicate any dependence and the average values can be adopted.															

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