



**SLOVENSKI STANDARD**  
**SIST EN 12458:1999**

**01-oktober-1999**

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Surface active agents - Determination of stability in hard water

Grenzflächenaktive Stoffe - Bestimmung der Beständigkeit gegenüber hartem Wasser

Agents de surface - Détermination de la stabilité a l'eau dure

**Ta slovenski standard je istoveten z: EN 12458:1999**

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**ICS:**

71.100.40 Površinsko aktivna sredstva Surface active agents

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EUROPEAN STANDARD

EN 12458

NORME EUROPÉENNE

EUROPÄISCHE NORM

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Descriptors: surfactants, tests, measurements, stability, water hardness

English version

## Surface active agents - Determination of stability in hard water

Agents de surface - Détermination de la stabilité à l'eau  
dureGrenzflächenaktive Stoffe - Bestimmung der Beständigkeit  
gegenüber hartem Wasser

This European Standard was approved by CEN on 25 January 1999.

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, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

**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 August 1999, and conflicting national standards shall be withdrawn at the latest by August 1999.

Annex A is informativ.

**Endorsement notice.**

The text of the International Standard ISO 1063:1974 was approved by CEN/TC 276 as a European Standard with the following modifications as given below :

- a) change of units "meq" to "mmol/l" ;
- b) inclusion of an informative Annex A ;
- c) text editorially revised.

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.

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## Introduction

A knowledge of the stability of surface active agents in hard water is of great importance for all the applications of these substances, since the precipitates obtained with hard water can reduce their effectiveness in practice.

The tests specified in this document are carried out with an aqueous solution of calcium chloride of known hardness.

In certain cases, it can be necessary to use other ions that give rise to hardness in water (see test report).

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## 1 Scope

This European Standard specifies a method of assessing the stability in hard water of surface active agents which are readily soluble at ambient temperature or slightly higher temperatures.

This method is applicable to surface active agents soluble in water at 20 °C. It can be extended to those which are soluble at approximately 50 °C.

## 2 Normative 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 12829, *Surface active agents - Preparation of water with known calcium hardness (ISO 2174:1990 modified)*.

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

ISO 835-2, *Laboratory glassware - Graduated pipettes - Part 2: Pipettes for which no waiting time is specified*.

## 3 Terms and Definitions

For the purposes of this standard, the following definitions apply:

### 3.1

#### hardness of water

the hardness of water is due to the presence of soluble alkaline earth compounds and especially of calcium salts ; it is expressed in millimoles of calcium (II) ions per litre

### 3.2

#### stability of a surface active agent in hard water

the stability of a surface active agent in hard water is determined by the solubility of the compounds formed by ion exchange between the surface active agent and the calcium ions, or by modification or the colloidal state by ionic forces, salt effect, pH value, etc.

## 4 Principle

Mixing of a solution of the surface active agent in differing concentrations with hard water solutions of differing known calcium hardness.

After leaving the solutions to stand in specified conditions, observation of their appearance, i.e. clearness, opalescence, cloudiness or precipitation.

## 5 Reagents

All reagents shall be of recognized analytical quality and the water used shall comply to grade 3 in accordance with EN ISO 3696.

### 5.1 Hard water solutions, prepared according to EN 12829.

- Solution S<sub>1</sub>, of calcium hardness 3 mmol (= 120,24 mg) of calcium (II) ions per litre ;
- Solution S<sub>2</sub>, of calcium hardness 4,5 mmol (= 180,36 mg) of calcium (II) ions per litre ;
- Solution S<sub>3</sub>, of calcium hardness 6 mmol (= 240,48 mg) of calcium (II) ions per litre.

## 6 Apparatus

Ordinary laboratory apparatus and glassware, together with the following :

**6.1 Fifteen test tubes**, for example 30 mm in diameter and 200 mm long, graduated at 50 ml.

Experience has shown that flat-bottomed test tubes are preferable, as they make it easier to observe cloudiness or precipitates.

**6.2 Pipette**, 5 ml, graduated at every 0,05 ml, complying with ISO 835-2.

**6.3 Thermostatically controlled water bath** for measurements carried out above 20 °C.

## 7 Procedure

### 7.1 Preparation of test solution

Prepare a stock solution of 50 g of the surface active agent in 1 000 ml of water at 20 °C. If the products are not readily soluble at 20 °C, prepare the solution at 50 °C. This temperature shall be stated in the test report.

### 7.2 Determination

Using the pipette (6.2), transfer 5,0 ml of the test solution (7.1) to one of the test tubes (6.1) and add the hard water solution S<sub>1</sub> (5.1) to give a volume of 50 ml.

NOTE Formation of foam can be troublesome and should be avoided when mixing the test solution with the hard water solution.

To achieve this, close the test tube containing the mixture with the hand or a bung, turn it slowly upside down and bring it back slowly to the original position. This operation shall take 1 s; repeat it ten times.

Leave the test tube to stand for more than 1 h but less than 2 h at  $(20 \pm 2)$  °C and examine at this temperature for precipitates, cloudiness or opalescence. In the case of a solution which changes with time (soap solution for example), the age of solution to the nearest 5 min, shall be stated in the test report. If it appears that the solubility of the calcium salts increases with temperature, carry out the test at  $(50 \pm 3)$  °C and make the observation at this temperature.

Carry out the determination similarly with 2,5 ml, 1,2 ml, 0,6 ml and 0,3 ml of the test solution (7.1).

Proceed similarly with identical volumes of the test solution (7.1) and with the hard water solutions S<sub>2</sub> and S<sub>3</sub> (5.1).

### 7.3 Scoring

Allot to the result of each determination a score number in accordance with the numbering system in Table 1.

**Table 1 - Score number corresponding to the appearance of the liquid**

Appearance of the liquid	Score number (unit value)
Clear	5
Opalescent	4
Cloudy	3
Slight precipitate	2
Heavy precipitate	1

In case of doubt between the score numbers (for example, cloudiness and slight precipitate), choose the less favourable score number.

A liquid which is not clear, but through which objects can be seen, is regarded as opalescent.

A liquid which is not clear, and through which objects cannot be seen, is regarded as cloudy.

**NOTE** Although solutions in hard water of certain commercial surface active agents containing insoluble inorganic products are perfectly transparent, small quantities of crystals can appear at the bottom of the test tube or on the surface of the liquid. These crystals are entirely different from the colloidal precipitates arising from hardness of the water and it should be necessary to centrifuge the solution, before testing, until it is clear.

## 8 Expression of results

### 8.1 Mean stability

In general, a surface active agent is given a single stability figure which represents the "mean stability".

Add the 15 unit values obtained as described in 7.3 to obtain the total value, and calculate the mean stability, as shown in Table 2.

Table 2 - Mean stability

Sum of the 15 unit values	Mean stability
15 to 18	"one"
19 to 37	"two"
38 to 56	"three"
57 to 74	"four"
75	"five"

### 8.2 Differential stability

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In some cases, however, it can be useful to note the stability of a surface active agent in hard water in terms of water hardness.

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For each of the three solutions S<sub>1</sub>, S<sub>2</sub> and S<sub>3</sub>, and in increasing order of hardness, add the five unit values obtained and determine the partial stability for each solution as shown in Table 3.

Table 3 - Differential stability

Sum of the five unit values for each sample of hard water	Partial stability	
5 or 6	one	= $\bar{1}$
7 to 12	two	= $\bar{2}$
13 to 18	three	= $\bar{3}$
19 to 24	four	= $\bar{4}$
25	five	= $\bar{5}$

Three figures are thus obtained, expressing the stability for each of the three hardness, 3 mmol/l, 4,5 mmol/l and 6 mmol/l, and characterizing the "differential stability". The latter is, for example,  $\bar{111}$  for the worst stability and  $\bar{555}$  for the best stability in hard water.

## 9 Test report

The test report shall include the following information :

- all details required for complete identification of the sample ;
- the reference to this European Standard ;



- c) the temperature of measurement ;
- d) mean stability at  $t$  °C : one to five ;
- e) differential stability at  $t$  °C :  $\overline{111} \dots \overline{555}$  ;
- f) any operation not included in this European standard, or regarded as optional, as well as any circumstances which may have influenced the results ;
- g) the date of the test.

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