



SLOVENSKI STANDARD
SIST EN 15647:2009
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Surface active agents - Determination of the dispersing effect of surfactants on powder

Grenzflächenaktive Stoffe - Bestimmung der Dispersionswirkung von Tensiden auf
Pulver

iTeh STANDARD PREVIEW

Agents de surface - Détermination de l'effet de dispersion des agents de surface sur les
poudres

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Ta slovenski standard je istoveten z: **EN 15647:2009**

ICS:

71.100.40 Površinsko aktivna sredstva Surface active agents

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EUROPEAN STANDARD

EN 15647

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ICS 71.100.40

English Version

Surface active agents - Determination of the dispersing effect of surfactants on powder

Agents de surface - Détermination des forces de dispersion
des surfactants sur la poudre

Grenzflächenaktive Stoffe - Bestimmung der
Dispersionswirkung von Tensiden auf Pulver

This European Standard was approved by CEN on 29 November 2008.

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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 CEN Management Centre has the same status as the official versions.

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

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Foreword

This document (EN 15647:2009) 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of an identical text or by endorsement, at the latest by July 2009, and conflicting national standards shall be withdrawn at the latest by July 2009.

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.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, 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 the United Kingdom.

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EN 15647:2009 (E)

1 Scope

This European Standard specifies a method for the determination of the effectiveness of surface active agents to create and to stabilize a dispersion of pigment powder in water. It is applicable to all classes of surface active agents and formulations of surface active agents. The method can also be applied analogously to other powders.

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.

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

dispersing effect

dispersing power of a surfactant on powder

effectiveness of a surfactant to produce and to stabilize a solid-liquid dispersion

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4 Principle

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The dispersing effect is determined by the "degree of dispersion". The "degree of dispersion" is the amount of solid particles kept suspended by the surfactant at a defined distance from the bottom of a measuring cylinder after a defined time related to a homogeneous distribution.

10,00 g zinc oxide is added to a measuring cylinder and mixed with 50 ml of an aqueous solution of the surface active agent (6 g/l). The mixture is stirred with a defined stirring speed for 30 minutes. The dispersion is then diluted by adding 150 ml water and stirred again with the same speed for another 30 minutes. After standing for 3 hours, during which the zinc oxide particles are allowed to sediment, a 5 ml sample is taken at the 150 ml marking of the measuring cylinder and the oven-dry mass is determined. The degree of dispersion is the amount of the oven-dry mass in percentage of the maximum amount. The maximum amount is obtained if a homogeneous dispersion is formed and no sedimentation occurs.

An important aspect of the method is to use reproducible stirring conditions. This is achieved by using defined stirrer and vessel dimensions together with a stirring speed that is adjusted using the length of the conical vortex formed.

5 Reagents

WARNING — Your attention is drawn to the regulations covering the handling of hazardous substances. Technical, organisational and personal protection measures should be observed.

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

5.1 water of defined hardness and pH value

5.2 **zinc oxide** of defined quality, [CAS number : 1314-13-2] (or an other pigment powder)

6 Apparatus

Ordinary apparatus and the following:

- 6.1 **weighing papers**
- 6.2 **250 ml graduated flask** for the preparation of the surfactant solution
- 6.3 **measuring cylinder** to measure 150 ml of water
- 6.4 **50 ml transfer pipette**
- 6.5 **250 ml measuring cylinders** with an inner diameter of 3,5 cm
- 6.6 **5 ml transfer pipettes** with a good closing peleus ball or **piston pipettes**
- 6.7 **magnetic stirrers**
- 6.8 **stirring bars in cross form**, length 25 mm, volume 1,5 ml
- 6.9 **drying oven or automatic moisture analyzer**
- 6.10 **Petri pan or pan of the automatic moisture analyzer**
- 6.11 **analytical balance**, accurate to $\pm 0,001$ g
- 6.12 **pH meter**

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7 Sampling and preparation of the test sample

The test sample shall be prepared and stored in accordance with ISO 607.

8 Procedure

8.1 Preparation of the surfactant solution

(1,500 \pm 0,002) g of the surfactant to be tested is weighed into a 250 ml graduated flask. About 100 ml of water are added and the surfactant is dissolved by swirling the liquid.

NOTE For surfactants which are hard to dissolve sonification may be used.

After the surfactant is completely dissolved the graduated flask is filled up with water to 250 ml. The pH value is determined. The standard cannot be applied, if the pH of the surfactant solution is out of the range from 6 to 9.

8.2 Positioning of the cylinder

The stirring bar in cross form is put in the 250 ml measuring cylinder. The measuring cylinder is placed on the magnetic stirrer in a way that the stirring bar can rotate freely. The position of the measuring cylinder on the magnetic stirrer is marked.

EN 15647:2009 (E)**8.3 Adjustment of the stirrer velocity**

The stirring speed is determined as follows:

50 ml of the surfactant solution is pipetted into the 250 ml measuring cylinder. The stirring speed of the magnetic stirrer is now adjusted, so that the tip of the conical vortex, which is formed when the stirring speed is increased, just touches the magnetic bar. This stirring speed is never changed during the test.

8.4 Carrying out of the test

8.4.1 (10,00± 0,01) g zinc oxide are weighed with a weighing paper and put carefully into a dry 250 ml measuring cylinder.

NOTE The use of a second weighing paper as a funnel for the cylinder is recommended.

8.4.2 50 ml of the surfactant solution are pipetted into the measuring cylinder.

NOTE The flow time of the pipette has to be followed.

8.4.3 The dispersion is stirred for 30 minutes on a magnetic stirrer with the adjusted stirring speed accordingly to 8.3.

8.4.4 In a separate measuring cylinder 150 ml of water are measured and added after these 30 minutes of stirring to the zinc oxide dispersion.

8.4.5 The dispersion is stirred for a further 30 minutes with the stirring speed adjusted.

8.4.6 After these 30 minutes the stirring is stopped and the dispersion is allowed to stand without any disturbance for 3 hours.

8.4.7 After the 3 hours of standing a 5 ml sample is taken out with a transfer pipette with a hard-closing peplus ball or with a piston pipette exactly at the height of the 150 ml marking of the measuring cylinder.

NOTE It is very important that no liquid is soaked up when the pipette crosses the surface of the liquid. A soaking up is avoided by closing the pipette at the top with a good working peplus ball or by the use of a piston pipette.

8.4.8 The weight of the oven-dry 5 ml sample is determined as follows:

a) by drying the sample on a Petri pan in an oven at 80 °C until the weight is constant (deviation < 1 mg after 50 seconds)

or

b) by drying the sample with an automatic moisture analyser at a drying temperature of 80 °C until the weight difference is less than 1 mg for 50 seconds.

If the initial weight of the 5 ml sample differs from 5,00 g, the oven-dry mass has to be corrected to correspond to a sample weight of 5,00 g.

9 Calculation and expression of results

The degree of dispersion is the value of the oven-dry mass of the 5 ml sample of the dispersion as a percentage of the maximum value. The maximum value is 0,25 g (10,00 g, divided by the factor 200 ml/5 ml) and is obtained theoretically for a homogeneous dispersion of the zinc oxide. (For the calculation of the maximum value the oven-dry mass of the surfactant is neglected.)

The degree of dispersion (D), expressed in %, is calculated by the equation (1) :

$$D = \frac{m \times 100}{0,25} \quad (1)$$

where

m is the oven-dry mass of the dispersion sample, in grams.

At least 3 tests with zinc oxide and a blank test with the surfactant alone shall be done. The resulting values shall be compiled as shown in Table1 and the average calculated:

Table 1 – Example of Table of results

Name of the Surfactant:

Test no.	Initial weight of the 5 ml sample g	oven-dry mass g corrected to a sample weight of 5,00 g	oven-dry mass of the dispersion sample minus the oven-dry mass of the blank sample	degree of dispersion %
1				
2				
3				
average				

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10 Precision

10.1 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.

Typical precision data obtained in ring tests are given in Annex A.

10.2 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.

Typical precision data obtained in ring tests are given in Annex A.