BS EN 15647:2009

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

ICS 71.100.40

National foreword

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The UK participation in its preparation was entrusted to Technical Committee CII/34, Methods of test for surface active agents.

A list of organizations represented on this committee can be obtained on request to its secretary.

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 28 February 2009

ISBN 978 0 580 58331 5

Amendments/corrigenda issued since publication

Date	Comments

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15647

January 2009

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

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Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 15647:2009: E

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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of an identical text or by endorsement, at the latest by July 2009, and conflicting national standards shall be withdrawn at the latest by July 2009.

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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

4 Principle

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 ± 0,001 g

6.12 pH meter

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 ± 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.

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 peleus 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 peleus 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 moister 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				

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.

11 Test report

The test report shall include the following information:

a) all information necessary for the identification of the surfactant tested;

b) all information necessary for the complete identification of the zinc oxide (or another pigment powder), like the specific density and the particle size distribution

c) the quality of the water used: water hardness, pH and temperature

d) a reference to this European Standard (EN 15647)

e) the test results ;

f) details of any operation not specified in this European Standard or in the European Standards to which reference is made, and any operations regarded as optional, as well as any incidents like to have affected the results.

Annex A
(informative)

Statistical and other data derived from the results of interlaboratory tests

The data for the repeatability and reproducibility limits of this method are the results of inter laboratory tests carried out by CESIO (Comité Européen des agents de Surface et de leurs Intermédiaires Organiques) in 2005 and 2006. The evaluation of the laboratory test was performed in accordance with ISO 5725-2.

The tested samples were the following :

— sample A : Tamol NH 7519;

— sample B : Baykanol SL.

Table A.1 - Interlaboratory test of samples

Designation	Sample A	Sample B
Number of participating laboratories	7	8
Number of accepted test results	21	24
Mean value (w) (g/100 g)	80,4	77,5
Repeatability standard deviation (s_r)	1,95	1,39
Repeatability coefficient of variation	2,4 %	1,8%
Repeatability limit (r) ($2{,}8 \times s_r$)	5,5	3,9
Reproducibility standard deviation (s_R)	4,29	2,45
Reproducibility coefficient of variation	5,3 %	3,2%
Reproducibility limit, (R) ($2{,}8 \times s_R$)	12,1	6,9

Bibliography

[1] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results. Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

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

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Headquarters 389
Chiswick High Road,
London, W4 4AL, UK
Tel +44 (0)20 8996 9001
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