SLOVENSKI STANDARD
SIST EN 14371:2005

01-april-2005

Površinsko aktivne snovi - Ugotavljanje penjenja in stopnje penjenja - Metoda preskušanja s kroženjem

Surface active agents - Determination of foamability and degree of foamability - Circulation test method

Grenzflächenaktive Stoffe - Bestimmung der Schäumfähigkeit und des Verschäumungsgrades - Zirkulations-Prüfverfahren

Agents de surface - Détection de l'aptitude au moussage et du taux de moussage - Méthode par circulation

Ta slovenski standard je istoveten z: **EN 14371:2004**

ICS:

71.100.40 Površinsko aktivna sredstva Surface active agents

SIST EN 14371:2005 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14371

September 2004

ICS 71.100.40

English version

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

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Contents

Foreword

This document (EN 14371:2004) 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of an identical text or by endorsement, at the latest by March 2005, and conflicting national standards shall be withdrawn at the latest by March 2005.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Introduction

The foamability and foamability degree are specified by means of a circulation equipment, whereby the solution of the surface active agent is under fast circulation for 10 min. During this circulation a definite volume of foam is generated which is a typical characteristic for the surface active agent at a certain concentration and temperature. This volume is called "foamability“ and is recorded on a 0 ml to 1 500 ml scale and expressed in millilitres.

Additionally the degree of foamability is recorded in parallel on a 0 % to 300 % scale or calculated from the foamability.

A basic objective is to test within a time, a defined circulation speed and a concentration range in which the foam formation comes to a saturation volume. This saturation foam volume nearly represents maximum foamability and is characteristic for any surface active agent.

After the circulation has been stopped, the destabilisation of the foam and the time in which half volume of foam is collapsed are recorded. The half volume collapsing time $t_{v/2}$ indicates the foam stability of the surface active agent.

A foamability profile versus temperature can be achieved by using single isothermal measurements (Method M 1) or by using a new continuous method (Method M 2), where the temperature is changed (increased) during the test. It was been found that the saturated volume quickly follows the temperature changes during circulation.

1 Scope

This document specifies a method for the determination of foamability and degree of foamability of surface active agents by means of a circulation equipment, whereby the solution of the surface active agent is under fast circulation.

The method is applicable to many surface active agents, especially for low and medium foaming surface active agents and products containing surface active agents.

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.

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

EN ISO 4788, *Laboratory glassware – Graduated measuring cylinders*(ISO/DIS 4788:2003)

3 Principle

A gear pump recycles 500 g of the test solution, which is carefully controlled concerning the temperature, in a 2 000 ml jacked cylinder and sprays it in a bypass system through a nozzle onto the surface of the liquid. During the test under high flow rate (200 l/h) a characteristic constant „saturated“ volume is formed which cannot significantly be increased by any increase of the circulation rate.

The volumes are measured either by visual recording or other equivalent recording auxiliaries like electronic foam height recording systems.

The additionally created foam, starting from a 500 g (ml) volume, is recorded each 30 s. In the time interval between 5,5 min and 10 min where a stable foam phase is observed, the average volume is determined. The foamability at the concentration c and the temperature T, $F_{(c,\ T)}$, results from it as follows:

$$F_{(c,\ T)} = \Sigma V_{s\ (5,5\ \text{min}...10\ \text{min})} / 10 = 0\ \text{ml} \ldots 1\ 500\ \text{ml}$$

where

$\Sigma V_{s\ (5,5\ \text{min}...10\ \text{min})}$ is the sum of the recorded, saturated volumes at 5,5 min; 6 min; 6,5 min; 7 min; 10 min.

Finally the generated volume (= foam) can be compared with the starting volume and is expressed or recorded at a scale as degree of foamability at the concentration c and the temperature T, $F_{D(c,\ T)}$:

$$F_{D(c,\ T)} := F_{(c,\ T)} / 5 = 0\ \% \ldots 300\ \%$$

Finally, having stopped the circulation the collapsing phase is recorded as the half volume of foam collapsing time $t_{v/2}$ expressed in seconds.

This indicates the stability of the foam and is an optional parameter, which can be measured. This value can be determined by extrapolating from a graph or the collected data values or measured directly.

First such a concentration is chosen, that at 25 °C a volume of foam of about 700 ml to 1 200 ml can be reached under above conditions, so that good foam characteristics with good resolutions of the values can be achieved.

Two equivalent (recording) test methods are available:

— Method M 1: Isothermal measurement

The recording is done at a defined constant temperature, e.g. at 25 °C; other preferred temperatures for recording are 20 °C, 30 °C, 40 °C ... 80 °C. Finally a foamability profile is plotted using the foamability (saturated volume) V_s versus temperature or the degrees of foamability [%] V_s versus temperature. Typically two recordings under identical conditions are made and the average values are taken.

— Method M 2: Continuous measurement

After an isothermal starting phase (5 min at 20 °C) a saturated foam volume is found. Afterward during a continuous temperature increase of 1 °C/min between 20 °C to 80 °C, the foam volumes are recorded. The saturated foam volume quickly follows the temperature changes and the foamability V_s versus temperature can be graphically recorded.

The values shall be recorded at each degree.

4 Reagents

4.1 General

During the test use only reagents of recognized analytical grade and water complying with grade 1 in accordance with EN ISO 3696.

4.2 Laboratory cleaner, e.g. phosphate based surface active agents, with low foaming, for glassware, capable of removing surface active agents.

4.3 Reference surface active agent (RS 1), for calibration.

NOTE If doubts occur about the correct function with a foamability of $F_{(0,01\ \%,\ 25\ °C)}$ = (1 030 ± 100) ml, see 6.3.

5 Apparatus

5.1 Test equipment, consisting of:

5.1.1 2 l-jacked cylinder, with 3 scales:

Scale 1: 0 ml to 500 ml, for indicating the test volume; 500 ml start level = 0.

Scale 2: 0 ml to 1 500 ml for the generated foam.

Scale 3: 0 % to 300 % degrees of foamability.

Inside dimensions shall be in accordance with ISO 4788. Dimensions of the outlet valve at the bottom are optional.

5.1.2 Gear pump, with a pump head flow rate of 0 l/h to 280 l/h where a rate of 200 l/h shall be adjustable.

5.1.3 Gear pump regulation unit.

5.1.4 Bypass system: Suction tube (8 mm), manometer (0 kPa to 60 kPa), tubes, nozzle (50 mm length, 5 mm diameter).

5.1.5 Electronic temperature sensor, arranged along and behind the suction tube, with a temperature indication either in a digital display or on the display at the thermostatic bath.

5.1.6 **Telescopic stand**, e.g. H-basement.

5.1.7 **Liftable pump suspension**, consisting of a supporting lever, pump on swinging lever and clamps.

5.1.8 **Thermostatic bath**, consisting either of a common thermostatic bath or a programmable thermostatic bath (preferably used for continuous recording).

5.2 **Beaker glass**, capacity 800 ml, for preparing the solutions.

5.3 **Water jet vacuum pump**, recommended for removing test solutions from the cylinder.

5.4 **Balance**, with an accuracy of 0,1 g.

5.5 **Stop watch**, with an accuracy of 0,1 s.

6 Test equipment

6.1 Installation of the test equipment

The individual parts (5.1.1 to 5.1.8) shall be assembled according to the supplier's instructions.

Install the telescopic stand, fix the suspension lever with the screw on the telescopic stand, at a height of 688 mm from the desk, fix the gear pump (5.1.2) on the swinging lever and fix the swinging lever above the supporting lever with a clamp (≈ 732 mm); shift both to a position (slight corrections are possible) where the suction tube can be installed on the pump head. The distance between suction tube and bottom of the cylinder shall finally be exactly 10 mm. Fix a clamp (≈ 445 mm) on the lower part of the telescopic stand, so that the whole upper part cannot be shifted lower than to the clamp. Now adjust this 10 mm distance between the suction tube and the bottom. The swinging lever can be moved upward together with the suction tube, if necessary, upward after a slight release at its fixation clamp. Then it is possible to remove the cylinder by lifting and moving the suction tube with the pump slightly to the operator.

Install the bypass with the manometer and the nozzle. The nozzle shall be in a position exactly central above the cylinder. Fix the electronic temperature sensor along and behind the suction tube with small plastic clamps and connect it to the thermostatic bath or to the device which indicates the internal temperature. Connect pump and regulation unit.

The arrangement of the test equipment is shown in Figure A.1.

6.2 Calibration of the pump and circulation flow

To determine the necessary flow rate of 200 l/h, it is helpful to measure the flow rate for at least three different pump settings, e.g. at 40, 60 and 80 intervals by regulating the pump control unit. Usually a setting of about 70 units gives a flow rate of 200 l/h.

Pour approximately 2 l of water into a 2 l calibrated beaker and circulate it at the desired setting. Take the mass of calibrated beaker, fill in during circulation until an amount of approximately 900 g from the water jet is reached. Time with the stopwatch (5.5) to the nearest 0,1 s. Read the mass of water caught in the beaker on the balance (5.4) to the nearest 0.1 g. Use the following short form of equation for the calculation of the flow rate, r_f:

$$r_f = \frac{m \times 3{,}6}{t} \qquad (1)$$

where

m is the mass of the water caught in the beaker, in grams;

t is the time, in seconds.

Using at least three different values, plot the most accurate calibration graph possible for pump delivery [l/h] against scale intervals. Extrapolate this graph to obtain the scale interval necessary for 200 l/h. If required, check the calibration at suitable time intervals.

Under standard conditions the manometer indicates a pressure of about 4 kPa.

If vibrations on the manometer are observed, residues in the pumps could be the reason. In this case clean the pump carefully. Should vibrations remain, exchange the gears for repair set.

6.3 Additional calibration with surface active agent

If there are any doubts about the correct function, the reference surface active agent (RS 1) (4.3) shall be used.

NOTE RS 1 was tested in a ring test at 25 °C and at a concentration of 0,01 % and gave a foamability of F = 1 030 ml, corresponding to a degree of foamability of F_D = 206 % (M 1). RS 1 is an EO/PO copolymer surface active agent.

7 Sampling and preparation of the test sample

Liquid samples can be used as delivered. They shall be free of abrasive solid particles to avoid damageto the gear pump.

Samples of solid surface active agents shall be dissolved in water to a 10 % (m/m) stock solution. The final calculation shall be made in respect to the original material.

The optimal concentration of the test solution shall be chosen from Table 1 such that at 25 °C about 700 ml to 1 200 ml foam are generated.

Usually the most suitable concentrations for low and medium foaming surface active agents are 0,05 % and 0,01 %.

Concentration steps given in Table 1 are recommended in order to reach a better reproducibility of the test results between different users and laboratories.

Table 1 — Recommended mass of surface active agent at different concentration steps for start volumes of 500 g each

Concentration range					
Normal range				Limited range	
		Preferred range			
0,50 %	0,10 %	0,05 %	0,01 %	0,005 %	(0,001 %)
2500 mg	500 mg	250 mg	50 mg	25 mg	5 mg

Weigh the requested amount of surface active agent (= x mg) in a glass beaker, dissolve with distilled water, transfer to a 500 ml volumetric flask and make up to volume.

8 Procedure

8.1 Settings of the test equipment

Check the following:

a) internal temperature of the test solution;

b) distance of the suction tube to the bottom : 10 mm;

c) central position of the nozzle above the cylinder;

d) manometer pressure : about 4 kPa;

e) nozzle : diameter 5 mm, length 50 mm;

f) distance nozzle to surface : about 430 mm;

g) concentration of test solution : see Table 1.

8.2 Cleaning of the test equipment and preparation of the test

Before analysing any new surface active agent, rinse the cylinder (5.1.1) with a cleaner (e.g. at a concentration of 0,5 %) by short circulation. Afterward rinse with water. Determine the foamability of the solution, remove the solution with the water jet vacuum pump (5.3) or via the outlet valve and then rinse with water.

NOTE If a specific surface active agent shows the tendency to be adsorbed to the glass (e.g. quats etc.), first a blank test run should be made with the test solution. However this result is not yet reliable, because some surface active agents with strong adsorbance behaviour are fixed onto the glass and can influence the following measurements. It has been found, that a "natural wetted“ glass surface gives more precise values, than glass pre-wetted with the cleaner only.

Rinse shortly with water, empty the cylinder (5.1.1) and proceed with the measurements.

8.3 Determination of foamability as example with "surface active agent A“

8.3.1 General

The following determination is demonstrated with the help of an example that is the "surface active agent A". This sample is a short chain non-ionic silicone surfactant (wetting agent) with high adsorption properties.

If there is not a satisfying congruency between the values measured according to Method M 1 (see 8.3.2) and Method M 2 (see 8.3.3), the values determined according to Method M 1 (isothermal method) shall be preferred due to their longer measuring time.

8.3.2 Isothermal, discontinuous Method M 1

Adjust the thermostatic bath (5.1.8) which is connected with the cylinder (5.1.1) to the preselected temperature, preferably at 25 °C. Pour carefully the externally prepared and pre-heated test solution into the cleaned cylinder and wait until the solution has reached the correct measuring temperature.

Start the gear pump (5.1.2) and record the foam volume in intervals of 30 s. Usually at least after 5 min a relatively saturated constant foam level can be observed. Continue the circulation for 10 min. Then stop the gear pump and record the collapsing behaviour for further 5 min (minimum) to obtain additional information about the foam stability. Determine the mean value of 10 readings recorded between 5,5 min and 10 min and express it as foamability *F* in millilitres.

The mean values of foamability *F* obtained from surface active agent A are given in Table 2.