BRITISH STANDARD

BS EN
13996:2002

Surface active agents — Foaming power and antifoaming power — Turbine stirring method

The European Standard EN 13996:2002 has the status of a British Standard

ICS 71.100.40

National foreword

This British Standard is the official English language version of EN 13996:2002.

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:

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

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Surface active agents - Foaming power and antifoaming power - Turbine stirring method

Agents de surface - Pouvoir moussant et pouvoir antimoussant - Méthode d'agitaion par turbine

Grenzflächenaktive Stoffe - Schäumvermögen und Entschäumerwirkung - Verfahren mittels Turbinenrührgerät

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Contents

Foreword

This document (EN 13996:2002) 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of an identical text or by endorsement, at the latest by April 2003 and conflicting national standards shall be withdrawn at the latest by April 2003.

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

1 Scope

This European Standard specifies a method for measuring the foaming power of a surface active agent and the antifoaming power of a defoamer with regard to a foaming solution.

The method is applicable to all surface active agents and particularly to low foaming products and antifoaming surface active agents.

However, measurement of the foaming power of solutions of readily hydrolyzable agents does not give valid results, as the hydrolysis products gather in the films of liquid and affect the persistence of the foam.

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 (including amendments).

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 607, *Surface active agents and detergents - Methods of sample division*.

ISO 1042, *Laboratory glassware - One-mark volumetric flasks*.

ISO 4788, *Laboratory glassware - Graduated measuring cylinders*.

3 Terms and definitions

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

3.1
foam
mass of gas cells separated by thin films of liquid and formed by the juxtaposition of bubbles, giving a gas dispersed in a liquid

3.2
foaming power
ability to produce foam

NOTE In this European Standard, foaming power is characterised by the volume of foam obtained immediately and the evolution of this volume during the 15 min following its formation, under specific experimental conditions.

3.3
foaming agent
substance which, when introduced into a liquid, confers on it an ability to form foam

3.4
antifoaming agent
defoamer
substance which prevents the formation of a foam or considerably reduces foam persistence

NOTE Antifoaming power is defined in relation to defined solutions of reference foaming agents such as saponin or nonylphenol with 10 ethylene oxide molecules (NP 10 EO).

In this European Standard, antifoaming power is characterised by the evolution of the volume of foam obtained immediately and the stability of this volume during the 15 min following its formation, as a function of the defoamer content of the reference foaming solution, under specific experimental conditions. Temperature is a significant parameter.

4 Principle

A foam is produced by stirring a surface active agent solution, or a reference foaming solution containing a defoamer, using a turbine stirrer.

The volume of foam obtained immediately is measured and the evolution of this volume during the 15 min following its formation is monitored to determine the foaming power of the surface active agent, or the defoaming power of the defoamer with regard to the reference foaming solution.

NOTE By varying the stirring methods, (for example by changing the stirrer, rotating speed, the presence of counterblades, baffle, the stirring duration) the procedure can be modified to give results for a wide range of applications.

5 Reagents

5.1 Water, grade 3, in accordance with EN ISO 3696.

5.2 Reference foaming agent, either nonylphenol 10 EO, with a cloud temperature of (64 ± 2) °C or saponin, analytical grade.

6 Apparatus

Ordinary laboratory apparatus and the following.

NOTE Perfect cleanliness of the apparatus is essential for the success of the test. Glassware can be cleaned by leaving it in contact with a mixture of chromic acid solution and sulfuric acid solution. The apparatus should always be rinsed with a small quantity of the solution under test.

6.1 Laboratory high speed stirrer[1)], with continuously variable speeds ranging from 50 min^{-1} to 3 500 min^{-1}, electronically controlled.

6.2 Centripetal blade stirrer[2)], with 40 mm diameter.

6.3 Baffle, (10 10) mm.

6.4 Stainless steel beaker, 2 l capacity with a height of 190 mm and diameter of 120 mm.

6.5 Thermometer, capable of measuring a range from 10 °C to 40 °C to the nearest 0,5 °C.

6.6 Timer, stopwatch or clock, capable of measuring to the nearest 2 s.

6.7 Tachometer, suitable for measuring the speed of the centripetal blade stirrer.

6.8 Graduated measuring cylinders, capacity 2 000 ml, in accordance with ISO 4788.

6.9 Volumetric flask, capacity 1 000 ml, in accordance with ISO 1042.

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1) Turbotest Compact 10-44 from Rayneri supplied by VMI (F.85601 Montaigu, France) is an example of suitable apparatus available commercially. This information is given for the convenience of users of this standard and does not constitute an endorsement by CEN of this apparatus.

2) Centripetal blade stirrer from Rayneri supplied by VMI (F.85601 Montaigu, France) is an example of suitable apparatus available commercially. This information is given for the convenience of users of this standard and does not constitute an endorsement by CEN of this apparatus.

6.10 Water bath, capable of maintaining a temperature of (20 ± 1) °C.

6.11 Stand, comprising a base and vertical rod, with an attachment to hold the laboratory high speed stirrer (6.1) and the centripetal blade stirrer (6.2) having an adjustable laboratory jack to support the stainless steel beaker (6.4).

7 Sampling

Prepare and store the laboratory sample in accordance with ISO 607.

8 Preparation of test solutions

8.1 Test solution for the foaming power test

Prepare from the surface active agent laboratory sample an aqueous solution of the material at its working strength, using either water (5.1) saturated with air by bubbling, or water of known hardness in accordance with EN 12829, previously equilibrated at the test temperature of (20 ± 1) °C.

NOTE It is necessary to mix very gently to prevent the formation of foam. The test temperature can be varied provided that the actual temperature is included in the test report.

Keep this test solution in the water bath (6.10) at the test temperature without stirring until the test is carried out.

The age of the test solution at the time of the measurement shall be not less than 30 min and not greater than 2 h.

The conditions of the preparation of the test solution (hardness of water, temperature) shall be included in the test report.

8.2 Test solutions for the antifoaming power test

8.2.1 Reference foaming solution

Prepare a 1 g/l reference foaming solution of the reference foaming agent (5.2), using either water (5.1) saturated with air by bubbling, or water of known hardness in accordance with EN 12829, previously equilibrated at the test temperature of (20 ± 1) °C. Mix very gently to prevent the formation of foam. Record the reference foaming agent used in the test report.

Keep the reference foaming solution in the water bath (6.10) at the test temperature. The reference foaming solution shall not be kept more than one day.

8.2.2 Test solutions of defoaming agent

Prepare test solutions of the defoaming agent with a range of defoamer contents, by pasting and then dissolution or dispersion in the reference foaming solution (see 8.2.1).

NOTE This mixture should be stable (limpid solution or stable dispersion). If the defoamer is insoluble or the dispersion is unstable, the defoamer should be directly introduced into the stainless steel beaker (6.4), at the start of the stirring.

The mixture can be made at a lower temperature than the chosen test temperature. In that case the 900 g of solution shall be brought up to the test temperature in the stainless steel beaker (6.4) using the water bath (6.10).

The age of the test solution at the time of measurement shall be not less than 30 min and not greater than 2 h.

The conditions of the preparation of the test solution (reference foaming agent, hardness of water, temperature, procedure used to introduce the defoamer) shall be included in test report.

9 Procedure

9.1 Adjustment of the apparatus

Set the height of the stand (6.11) using the laboratory jack, the position of the baffle (6.3) and the centripetal blade stirrer (6.2) so that it is possible to adjust the stainless steel beaker (6.4) rapidly as shown in Figure 1.

Fill the stainless steel beaker with 900 g of water, place it on the stand and adjust the height as shown in Figure 1. Start the laboratory high speed stirrer (6.1) and adjust its rotating speed to 2 000 min^{-1} using the tachometer (6.7). Stop the laboratory high speed stirrer (6.1), lower the beaker and discard the water.

9.2 Determination of foaming and antifoaming power

9.2.1 Foam formation

Carefully pour 900 g of the test solution for the foaming power test (8.1) respectively of the test solution for the antifoaming power test (8.2.2) into the stainless steel beaker (6.4), avoiding the formation of foam. Check the temperature and, if necessary, adjust it to (20 ± 1) °C, by returning it into the water bath (6.10) to equilibrate.

Place the beaker on the stand, adjusting the position as shown in Figure 1.

Simultaneously turn on the laboratory high speed stirrer (6.1) at 2 000 min^{-1} and start the timer (6.6). Stop the stirring after 5 min ± 10 s.

9.2.2 Foam measurement

Pour the solution and the foam rapidly from the beaker into a graduated cylinder (6.8). by allowing them to fall at the 1 600 ml mark and slide down the glass wall as shown in Figure 2. Restart the timer.

Then measure and record the upper level of the foam (air/foam interface) and the lower level of the foam (foam/solution interface) as well as the foam volume to the nearest 1ml at 0 min, 1 min, 2 min, 3 min, 5 min, 10 min and 15 min (each to the nearest 5 s) from the end of the stirring phase.

At the end of the test, gently agitate the graduated cylinder with a circular movement and record the residual foam volume.

10 Calculation and expression of results

10.1 Foaming test

Report the foam volume and the upper and lower levels of foam measured at 0 min, 1 min, 2 min, 3 min, 5 min, 10 min and 15 min.

Plot the graph of total foam volume in millilitres against time in minutes and the graph of upper and lower levels in millilitres versus time in minutes. An example of the graphs is given in Figure 3.

10.2 Antifoaming test

Report the foam volume and the upper and lower levels of foam measured at 0 min, 1 min, 2 min, 3 min, 5 min, 10 min and 15 min for each defoamer concentration.

Plot the graph of total foam volume in millilitres against time in minutes for each defoamer concentration.

The foaming power is given by the delimited surface between the curve and the x axis between 1 min and 15 min, calculated by means of the trapezium method (see Figure 4) in millilitres per minute.

NOTE The smaller the surface, S_c, is, the better is the antifoaming power.

Calculate the antifoaming power efficiency, E, as percent, using the following equation.

$$E = \frac{S_o \quad S_c}{S_o} \quad 100$$

where

S_o is the level of the surface using the reference foaming agent alone;

S_c is the level of the surface using the reference foaming agent with a given concentration of defoamer.

The antifoaming efficiency, in percent, against concentration can also be plotted as shown in Figure 5.

11 Precision

11.1 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.

The ring test results are shown in Tables A.1 and A.2.

11.2 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.

The ring test results are shown in Tables A.1 and A.2.

12 Test report

The test report shall contain the following information:

a) all information necessary for the complete identification of the sample;

- ¾ for the foaming test: nature and concentration of the surface active agent;
- ¾ for the antifoaming test: nature and concentration of the reference foaming agent solution and nature and tested concentrations of the defoamer;

b) the method used by reference to this European Standard;

c) the results obtained as specified in clause 10;

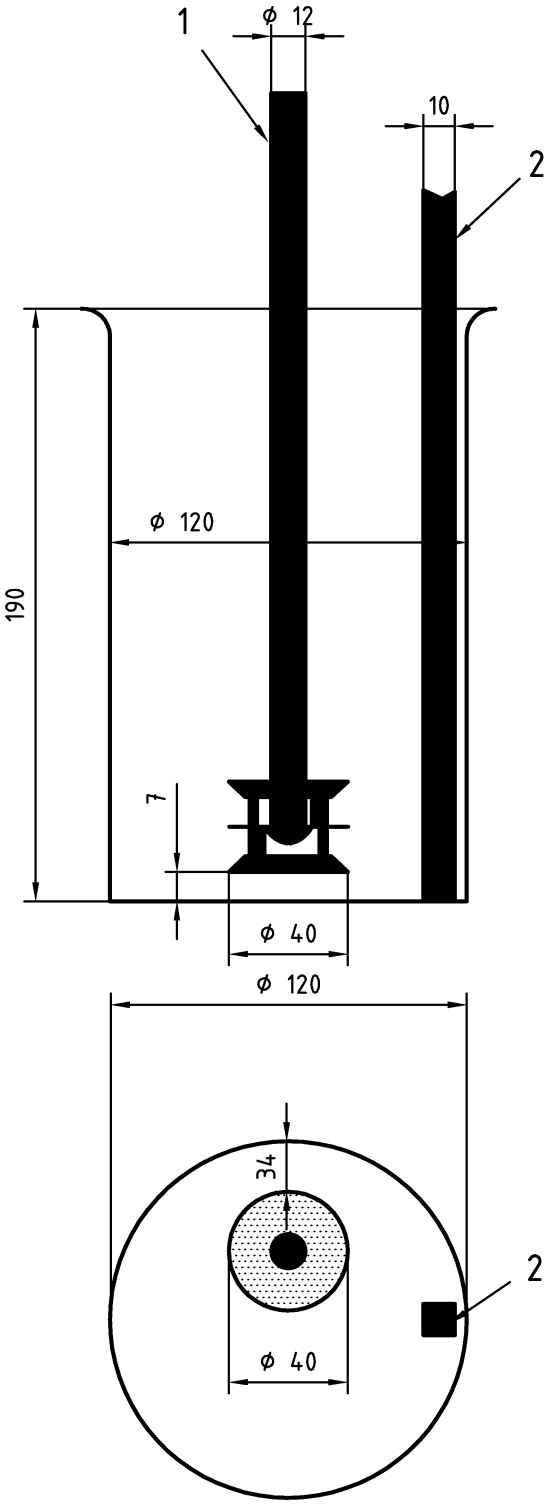
d) the test parameters, including:

- ¾ the date of the test;
- ¾ the test temperature, in degrees Celsius;
- ¾ the hardness of the water used, expressed in milligram equivalents of calcium (II) ions per litre, if this differs from water according to 5.1;
- ¾ age of the surface active agent solution;

- ¾ nature of the stirrer, if it differs from a centripetal blade stirrer;
- ¾ stirring speed;
- ¾ stirring duration;
- ¾ presence or absence of a baffle;

e) details of any operations not specified in this European Standard and any operations regarded as optional, and any incidents likely to have affected the results.

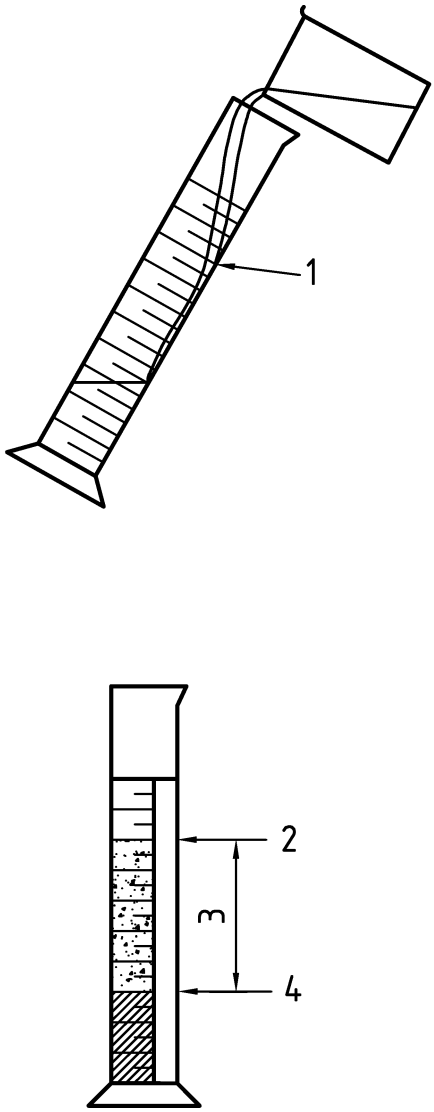
Dimensions in millimetres



Key

1 Centripetal blade
2 Baffle

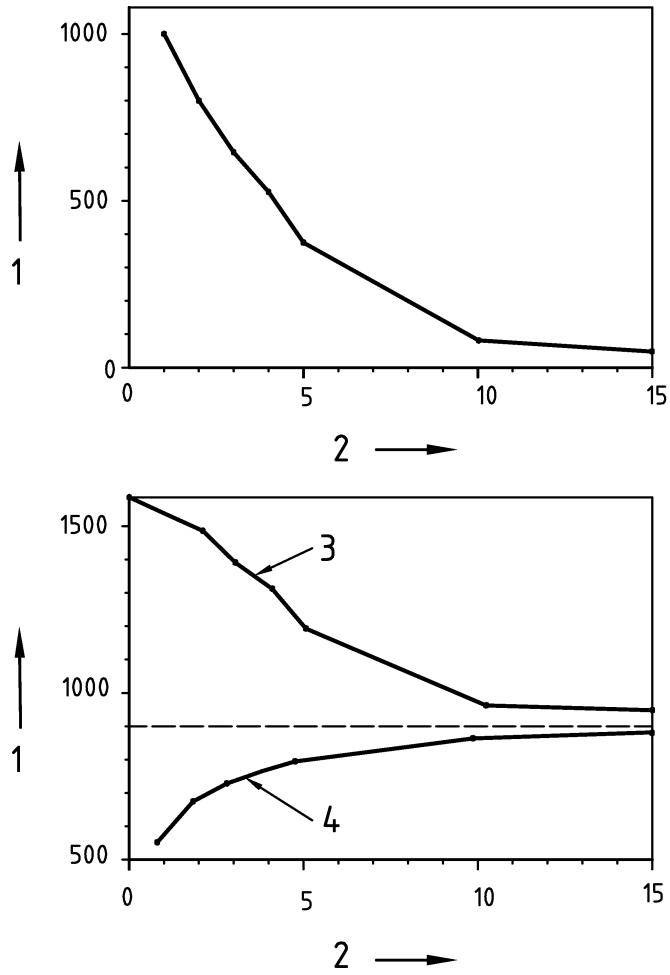
Figure 1 — Foaming and antifoaming power by turbine stirring apparatus



Key

1 Level 1 600 ml
2 Upper level of the foam in millilitres
3 Foam volume in millilitres
4 Lower level of the foam in millilitres

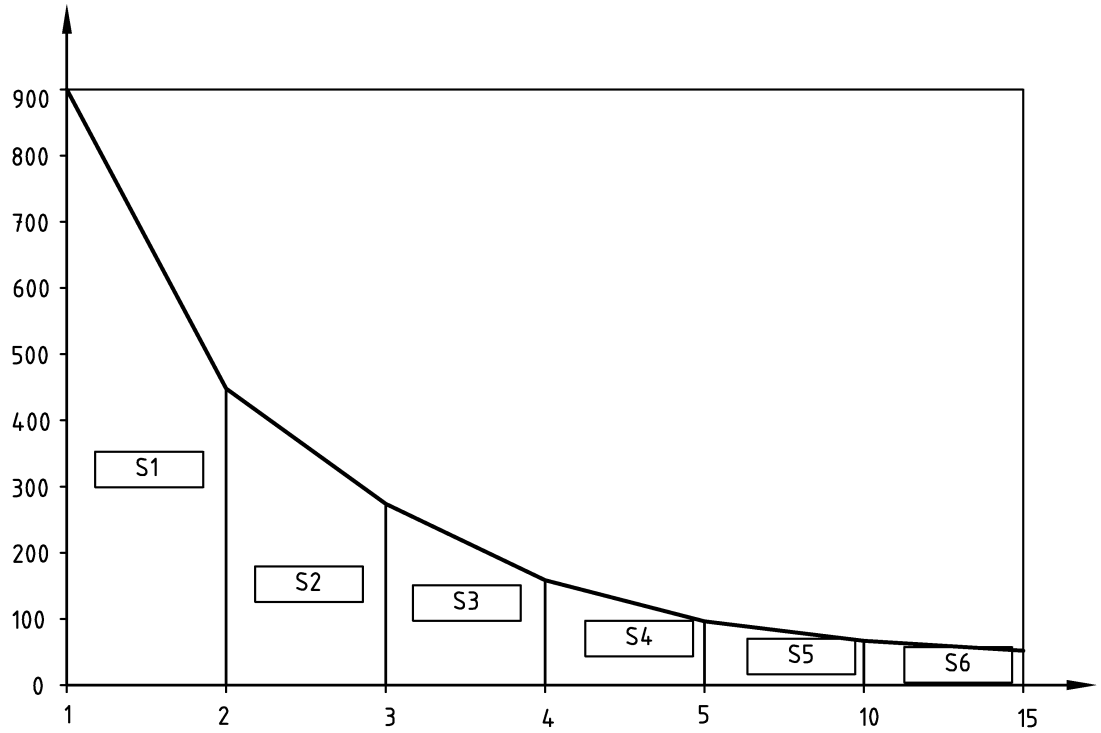
Figure 2 — Operation steps for measuring the foam volume



Key

1 Foam volume in millilitres
2 Time in minutes
3 Upper level of the foam in millilitres
4 Lower level of the foam in millilitres

Figure 3 — Foam volume and upper and lower levels of foam as function of the time



Key

1 Foam volume in millilitres

2 Time in minutes

¾ S_1: Surface 1

¾ S_2: Surface 2

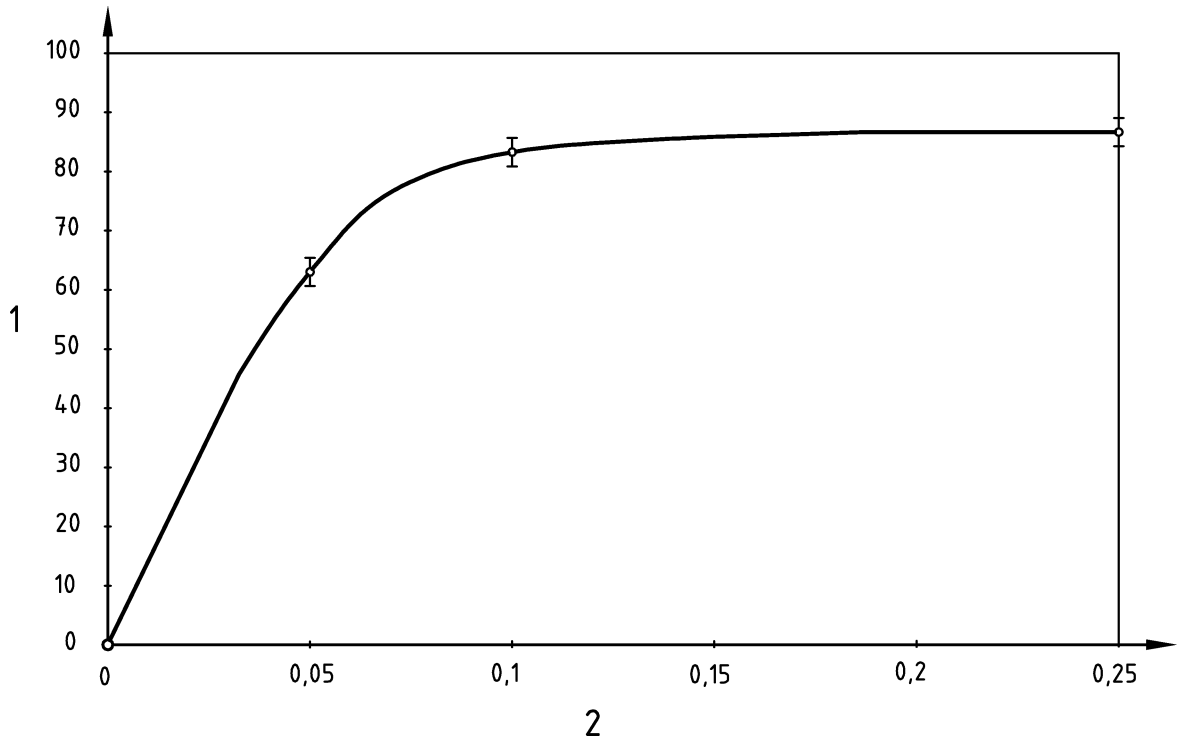
¾ S_3: Surface 3

¾ S_4: Surface 4

¾ S_5: Surface 5

¾ S_6: Surface 6

Figure 4 — Foaming power: $S = S_1 + S_2 + S_3 + S_4 + S_5 + S_6$



Key

1 Efficiency in percent

2 Concentration in grams per litre

Figure 5 — Antifoaming efficiency as function of the concentration

Annex A
(informative)

Ring test result

The ring test was carried out from CESIO/AISE in 1998. The symbols used in Tables A.1 and A.2 mean the following:

A = Nonylphenol 10 EO 1 g/l

B = Saponins 1 g/l

C = Plurafac® LF 231[3] or equivalent

D = Deterflo A 210[4] or equivalent

Table A.1 — Ring test results of the determination of foaming power

Designation	Sample A	Sample B	Sample C	Sample D
Number of participating laboratories	7	7	1	1
Number of not-eliminated laboratories	6	6	-	-
Number of values	6	6	4	4
Mean value w_s [S = surface delimited by the curves V = f (t)]	10 517	9 576	8 508	7 414
Repeatability standard deviation s_r	-	-	114	130
Repeatability limit r (r = 2,8 s_r)	-	-	319	365
Repeatability coefficient of variation %	-	-	1,34	1,76
Reproducibility standard deviation (s_R)	11	13	-	-
Reproducibility limit R (R = 2,8 s_R)	1138	1226	-	-
Reproducibility coefficient of variation, %	31,85	34,33	-	-

Table A.2 — Ring test results of the determination of antifoaming power

Designation	1 g/l C on A at 25 °C	1 g/l C on B at 25 °C	1 g/l D on A at 25 °C	1 g/l D on B at 25 °C
Number of laboratories	7	7	1	1
Number of not-eliminated laboratories	6	6	-	-
Number of values	6	6	4	4
Mean value w_E	59	84	60	84
Repeatability standard deviation s_r	-	-	0,29	2,07
Repeatability limit r (r = 2,8 s_r)	-	-	0,82	5,81
Repeatability coefficient of variation %	-	-	0,49	2,46
Reproducibility standard deviation (s_R)	2,07	1,59	-	-
Reproducibility limit R (R = 2,8 s_R)	5,80	4,45	-	-
Reproducibility coefficient of variation, %	3,51	1,89	-	-

3) Product from BASF AG, Germany.

4) Product from CECA SA / Division of ATOFINA, France.

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