BRITISH STANDARD

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Surface active agents — Determination of physical stability in alkaline and acid solutions

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ICS 71.100.40

National foreword

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English version

Surface active agents - Determination of physical stability in alkaline and acid solutions

Agents de surface - Détermination de la stabilité physique dans les solutions alcalines et acides

Grenzflächenaktive Stoffe - Bestimmung der physikalischen Stabilität in alkalischer und saurer Lösung

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Contents

Foreword

This European Standard (EN 14712:2005) 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of an identical text or by endorsement, at the latest by November 2005, and conflicting national standards shall be withdrawn at the latest by November 2005.

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Introduction

The stability of a surface active agent in alkaline and acid solutions is its physical stability during the defined time period, as judged by its appearance. The statement "the surface active agent A has an alkaline stability of *x* g NaOH per litre" means that the surface active agent A can be dissolved in a concentration of 10 g/l in an aqueous solution containing up to *x* g NaOH per litre to give a stable solution. Analogously the statement "the surface active agent A has an acid stability of *y* ml sulfuric acid per litre" means that the surface active agent A can be dissolved with a concentration of 10 g/l in an aqueous solution containing up to *y* ml sulfuric acid per litre (with a purity of 95 % to 97 %) to give a stable solution.

It should be noted that miscibility gaps can occur, i.e. the solution can be unstable at intermediate alkali or acid concentrations.

The definition of a stable solution depends on the application of the surface active agent or surface active agent formulation. Thus homogeneous opalescent or turbid solutions may be considered either stable or unstable.

1 Scope

This European Standard specifies a method for the determination of physical stability of surface active agents in alkaline and acid solutions. It is applicable to all classes of surface active agents and also to other substances or mixtures such as polymers, complexing agents and formulations of 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).*

3 Terms and definitions

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

3.1
alkaline stability
maximum mass concentration of sodium hydroxide (purity at least of 98 %) in grams per litre at which the surface active agent can be dissolved in the alkaline solution with a mass concentration of 1 % to yield a stable solution

3.2
acid stability
maximum volume concentration of sulfuric acid (purity 95 % to 98 %), in millilitres per litre, at which the surface active agent can be dissolved in the acid solution with a mass concentration of 1 % to yield a stable solution

3.3
other bases and acids
other bases and acids may be used. This should be described together with the purity in the test report

4 Principle

Aqueous solutions are prepared with increasing concentrations of sodium hydroxide or sulfuric acid respectively. Then the surface active agent to be tested is mixed in these aqueous solutions. The formulations are examined visually after 24 h of storage at ambient temperature (22 ± 3) °C to see whether a stable solution resulted or not. Clear homogeneous solutions are always considered stable, whereas phase separation indicates instability. Homogeneous opalescent or homogeneous turbid samples can be considered either stable or unstable depending on the surface active agent application.

5 Reagents

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

5.2 Sodium hydroxide, NaOH, analytical grade, preferably as an aqueous solution with a mass concentration of 30 % NaOH (this corresponds to 398 g NaOH (100 % active substance) in 1 l ; density ρ= 1,328 g/ml at 20 °C).

5.3 Sulfuric acid, H_2SO_4, analytical grade, preferably as an aqueous solution with a mass concentration of 25 % H_2SO_4 (density ρ = 1,18 g/ml at 20 °C).

6 Apparatus

6.1 General

Ordinary laboratory apparatus and the following:

6.2 Glass beakers or bottles with magnetic stirrer sticks, for storing and assessing the sample solutions. 125 ml screw-top jars are recommended.

NOTE Within the series the same type of beakers or bottles should be used.

6.3 Magnetic stirrer.

6.4 Beaker, for the preparation of the stock solution of the surface active agents.

6.5 Analytical balance, accurate to ± 0,01 g.

7 Procedure

7.1 General

An aqueous stock solution of the surface active agent to be tested shall be prepared with a mass concentration of 100 g/l. If surface active agent trade products are used, relate the mass concentration of 100 % active substance.

7.2 Alkaline stability

Pour and mix the corresponding amount of water (5.1) and of sodium hydroxide (5.2), preferably as a sodium hydroxide solution with a mass concentration of 30 % NaOH, into a series of beakers or bottles, for example 125 ml screw-top jars, (6.2) according to Table 1.

Table 1 — Series of solution of sodium hydroxide

Jar (6.2)	**Water** (5.1)	**Quantity of NaOH solution** (mass concentration of 30 %)		**Concentration of NaOH**
	g or ml	**g**	**ml**	**g/l**
A	87,5	3,3	2,5	10
B	80,0	13,3	10	40
C	70,0	26,7	20	80
D	60,0	40,0	30	120
E	50,0	53,3	40	160
F	40,0	66,7	50	200
G	30,0	80,0	60	240
H	20,0	93,3	70	280
I	10,0	106,7	80	320
J	0	120,0	90	360

Then pipette 10 ml or weigh 9,9 g of the stock solution of the surface active agent into each beaker, for example the 125 ml jars, to prepare a test solution of the surface active agent with a mass concentration of 10 g/l. (The density of the stock solution is normally about 0,993 g/ml at 20 °C). If the stock solution is inhomogeneous, do not

use it but fill the corresponding mass of the surface active agent calculated to 100 % active substance directly in the beakers.

Mix the test samples with the stirrer sticks on the magnetic stirrer (6.3) for about 5 min at about 400 min^{-1}. Allow the test samples to stand at ambient temperature (22 ± 3) °C for 24 h and then evaluate them. Gentle stirring or shaking is recommended, as slight phase separation is often difficult to detect otherwise. Turbid test samples shall be poured out in a second beaker to test, if any sediment was formed in the first beaker.

Evaluate the NaOH concentrations between the stable test samples and the unstable ones in a second series after testing this first series.

The volumes and equivalent mass concentrations of the components of the first and the second series are given in Table A.1.

NOTE The test may be carried out at other temperatures. If, for example, temperature controlled storage chambers are used, this should be noted in the test report.

7.3 Acid stability

Pour and mix the corresponding amount of water (5.1) and sulfuric acid (5.3) into a series of beakers or bottles, for example 125 ml screw-top jars, (6.2) according to Table 2.

Table 2 — Series of solution of sulfuric acid

Jar (6.2)	**Water** (5.1)	**Quantity of sulfuric acid solution (5.3)** (mass concentration of 25 %)		**Resulting volume concentration of sulfuric acid** (100 % a.s.)
	g or ml	**g**	**ml**	**ml/l**
A	89,6	0,5	0,4	1
B	88,0	2,4	2,0	5
C	86,0	4,7	4,0	10
D	82,0	9,4	8,0	20
E	74,0	18,9	16,0	40
F	66,0	28,3	24,0	60
G	58,0	37,8	32,0	80
H	50,0	47,2	40,0	100
I	42,0	56,6	48,0	120
J	34,0	66,1	56,0	140

Then pipette 10 ml or weigh 9,9 g of the stock solution of the surface active agent into each beaker, for example the 125 ml jars, to prepare a test solution of the surface active agent with a mass concentration of 10 g/l. (The density of the stock solution is normally about 0,993 g/ml at 20 °C). If the stock solution is inhomogeneous, do not use it but fill the corresponding mass of the surface active agent calculated to 100 % active substance directly in the beakers.

Mix the test samples with the stirrer sticks on the magnetic stirrer (6.3) for about 5 min at about 400 min^{-1}. Allow the test samples to stand at ambient temperature (22 ± 3) °C for 24 h and then evaluate them. Gentle stirring or shaking is recommended, as slight phase separation is often difficult to detect otherwise.

Turbid test samples shall be poured out in a second beaker to test, if any sediment has been formed in the first beaker.

Evaluate the sulfuric acid concentrations between the stable test samples and the unstable ones in a second series after testing this first series.

The volumes and equivalent mass concentrations of the components of the first and the second series are given in Table A.2.

NOTE The test may be carried out at other temperatures if, for example, temperature controlled storage chambers are used, this should be noted in the test report.

8 Expression of results

Use the score list according to Table 3 for the evaluation of the appearance of the test sample.

Table 3 — Evaluation of the test sample

Score	Appearance of the test sample
1	homogeneous clear
2	homogeneous opalescent
3	homogeneous turbid
4	macroscopic phase separation

A liquid which is not clear, but through which objects can be recognized, is rated as "opalescent".

An example is a liquid with 100 formazine nephelometric units.

A liquid which is not clear, and through which objects can not be recognized, is rated as “turbid”.

An example is a liquid with 1 000 formazine nephelometric units.

In some cases a phase separation of a test sample is not or hardly visible by the naked eye. Therefore clear test samples shall be stirred or shaken, as mentioned in 7.2 or 7.3. If the test sample now shows turbidity, it is rated as "macroscopic phase separation". A turbid test sample which shows a sediment when poured out of the beaker is rated as "macroscopic phase separation".

According to this score list, the alkaline stability of a surface active agent is the highest NaOH concentration, in grams per litre, of that solution which is stable. Analogously, the acid stability of a surface active agent is the highest sulfuric acid concentration, in millilitres per litre, of the solution which is stable. Clear homogeneous test samples are always considered stable, whereas phase separation indicates instability. Homogeneous opalescent or homogeneous turbid test samples may be considered either stable or unstable, depending on the surface active agent application. The definition of stability used shall be included in the test report.

9 Test report

The test report shall be made using the forms according to Annex A.

Annex A
(normative)

Forms for the test report

Table A.1 — Form for test report on the determination of alkaline stability of a surface active agent

Name of the sample :	Distinctive features
Tested in accordance with EN 14712	(e.g. miscibility gap) :
Name of the surface active agent :	Date :
Storage temperature :	Signature of the examinator :

Quantity of water	**Quantity of NaOH solution** (mass concentration **of 30 %)**		**Concentration of NaOH**	**Score of evaluation after 24 h**	
g or ml	**g**	**ml**	**g/l**	**result**	**remarks**
87,5	3,3	2,5	10		
85,0	6,7	5,0	20		
82,5	10,0	7,5	30		
80,0	13,3	10,0	40		
77,5	16,7	12,5	50		
75,0	20,0	15,0	60		
72,5	23,3	17,5	70		
70,0	26,7	20,0	80		
67,5	30,0	22,5	90		
65,0	33,3	25,0	100		
62,5	36,7	27,5	110		
60,0	40,0	30,0	120		
57,5	43,3	32,5	130		
55,0	46,7	35,0	140		
52,5	50,0	37,5	150		
50,0	53,3	40,0	160		
47,5	56,7	42,5	170		
45,0	60,0	45,0	180		
42,5	63,3	47,5	190		
40,0	66,7	50,0	200		
37,5	70,0	52,5	210		
35,0	73,3	55,0	220		
32,5	76,7	57,5	230		
30,0	80,0	60,0	240		

"to be continued"

Table A.1 *(concluded)*

Quantity of water	**Quantity of NaOH solution** (mass concentration of 30 %)		**Concentration of NaOH**	**Score of evaluation after 24 h**	
g or ml	**g**	**ml**	**g/l**	**result**	**remarks**
27,5	83,3	62,5	250		
25,0	86,7	65,0	260		
22,5	90,0	67,5	270		
20,0	93,3	70,0	280		
17,5	96,7	72,5	290		
15,0	100,0	75,0	300		
12,5	103,3	77,5	310		
10,0	106,7	80,0	320		
7,5	110,0	82,5	330		
5,0	113,3	85,0	340		
2,5	116,7	87,5	350		
0	120,0	90,0	360		
NOTE 10 ml or 9,9 g of the stock solution of the surface active agent with a mass concentration of 10 % are added additionally to each beaker (see Clause 7).					

Table A.2 — Form for the test report on the determination of acid stability of a surface active agent

Name of the sample :	Distinctive features
Tested in accordance with EN 14712	(e.g. miscibility gap) :
Name of the surface active agent :	Date :
Storage temperature :	Signature of the examinator :

Quantity of water	**Quantity of sulfuric acid solution** (mass concentration of 25 %)		**Volume concentration of sulfuric acid**	**Score of evaluation after 24 h**	
g or ml	**g**	**ml**	**ml/l**	**result**	**remarks**
89,6	0,5	0,4	1		
89,2	0,9	0,8	2		
88,8	1,4	1,2	3		
88,4	1,9	1,6	4		
88,0	2,4	2,0	5		
86,0	4,7	4,0	10		
85,2	5,7	4,8	12		
84,0	7,1	6,0	15		
82,0	9,4	8,0	20		
80,0	11,8	10,0	25		
78,0	14,2	12,0	30		
76,0	16,5	14,0	35		
74,0	18,9	16,0	40		
72,0	21,2	18,0	45		
70,0	23,6	20,0	50		
68,0	26,0	22,0	55		
66,0	28,3	24,0	60		
64,0	30,7	26,0	65		
62,0	33,0	28,0	70		
60,0	35,4	30,0	75		
58,0	37,8	32,0	80		
56,0	40,1	34,0	85		
54,0	42,5	36,0	90		
52,0	44,8	38,0	95		
50,0	47,2	40,0	100		
48,0	49,6	42,0	105		
46,0	51,9	44,0	110		
44,0	54,3	46,0	115		
42,0	56,6	48,0	120		
40,0	59,0	50,0	125		
38,0	61,4	52,0	130		
36,0	63,7	54,0	135		
34,0	66,1	56,0	140		

NOTE 10 ml or 9,9 g of the stock solution of the surface active agent with a mass concentration of 10 % are added additionally to each beaker (see Clause 7).

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