International Standard 6387

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Surface active agents — Determination of the power to disperse calcium soap — Acidimetric method (Modified Schönfeldt method)

Agents de surface — Détermination du pouvoir dispersant vis-à-vis du savon calcique — Méthode acidimétrique (Méthode de Schönfeldt modifiée)

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6387 was developed by Technical Committee ISO/TC 91, *Surface active agents*, and was circulated to the member bodies in April 1982.

It has been approved by the member bodies of the following countries :

Australia	Germany, F.R.	Netherlands
Austria	Hungary	Poland
Belgium	Iran	Romania
China	Ireland	South Africa, Rep. of
Czechoslovakia	Japan	Spain
Egypt, Arab Rep. of	Korea, Rep. of	Switzerland
France	Mexico	USSR

No member body expressed disapproval of the document.

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INTERNATIONAL STANDARD ISO/DIS 6387-1983 (E)

Surface active agents — Determination of the power to disperse calcium soap — Acidimetric method (Modified Schönfeldt method)

1 Scope

This International Standard specifies an acidimetric method of test for determining the minimum quantity of dispersing agent (surface active agent) required to maintain at least 95 % of the calcium soap present in complete dispersion for one hour.

NOTE — This method may also provide information on the amount of calcium soap partially dispersed by insufficient quantities of dispersing agents (surface active agents).

2 Field of application

This International Standard applies to all types of surface active agent insofar as these do not interfere during the acidimetric titration of calcium soaps. Alkaline inorganic salts such as phosphates, carbonates and silicates shall not be present.

3 Reference

ISO 2174, *Surface active agents — Preparation of water with known calcium hardness.*

4 Definition

For the purpose of this International Standard, the following definition is applicable :

power to disperse calcium soap : The quantity of soap, expressed in grams, which may be completely dispersed by 1 g of dispersing agent (surface active agent).

5 Principle

Preparation of a 0,5 % (m/m) aqueous soap solution, and after maintaining this at testing temperature for 24 h, taking an aliquot portion of this solution. Mixing of this aliquot portion with a dilute solution of the dispersing agent (surface active agent), and subsequently with a specified volume of water of known calcium hardness. Maintaining the mixture for 1 h at the testing temperature (the flocculated layer of calcium soap having reached the surface) and titrating the calcium soap present in an aliquot portion of the lower layer with a standard volumetric solution of hydrochloric acid in the presence of bromocresol green as indicator.

6 Reagents

During the analysis use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

6.1 Water of known calcium hardness, prepared as specified in ISO 2174.

6.2 Sodium oleate, 100 g/l solution.

Weigh, to the nearest 0,001 g, 92,78 g of oleic acid and dissolve it in 328,5 ml of sodium hydroxide solution, c(NaOH) = 1 mol/l. Allow to cool to ambient temperature. Transfer quantitatively the solution to a 1 000 ml one-mark volumetric flask and make up to the volume with water.

6.3 Hydrochloric acid, standard volumetric solution, c(HCl) = 0,01 mol/l.

6.4 Bromocresol green ($C_{21}H_{14}Br_4O_5S$), 1 g/l solution.

Dissolve 0,25 g of bis-3,3 (2-methyl-3,5 dibromo 4 hydroxy phenyl)-2,1 benzoxathiol-1,1 dioxide in 57,2 ml of sodium hydroxide solution, c(NaOH) = 0,01 mol/l. Transfer quantitatively the solution to a 250 ml one-mark volumetric flask and make up to the volume with water.

7 Apparatus

Ordinary laboratory apparatus and

7.1 Graduated measuring cylinders, of capacity 100 ml, with ground necks, complying with the requirements of ISO 4788.

7.2 Pipettes, of capacity 10 and 20 ml, complying with the specifications of ISO 648.

7.3 Thermostatically controlled bath, with a temperature range of 27 ± 0,5 °C to 40 ± 0,5 °C.

8 Procedure

8.1 Preparation of samples

8.1.1 Dilute soap solution

Transfer 50,0 ml of the sodium oleate solution (6.2) corresponding to 5,00 g of anhydrous soap into a 1 000 ml one-mark volumetric flask and make up to volume. Maintain the solution at the test temperature (27 °C or 40 °C) for a minimum of 24 h and not more than 48 h, before the test.

8.1.2 Solution of dispersing agent

Dissolve 1,00 g of the dispersing agent (surface active agent) (5,00 g if its dispersing power is low) in 1 litre of water and heat the solution to the test temperature.

8.1.3 Waters

Heat the water of known calcium hardness (6.1) and the water used for dilution to the test temperature.

8.2 Titration of soap solution

Using one of the pipettes (7.2), transfer 20,0 ml of the dilute soap solution (8.1.1) to a graduated cylinder (7.1) and make up to 100 ml with water.

Take 10,0 ml of this solution, add 3 drops of the bromocresol green solution (6.4) and titrate it with the hydrochloric acid solution (6.3) to a sharp change from blue to green.

8.3 Determination

Using one of the pipettes (7.2), transfer 20,0 ml of the soap solution (8.1.1) to a graduated cylinder (7.1). Add V_1 ml of the solution of the dispersing agent (8.1.2) and $(80 - V_1 - V_2)$ ml of the water (8.1.3). (V_2 is the number of millilitres of water of known calcium hardness required to give the hardness desired for the test.) Seal the cylinder with its ground glass stopper and mix by slowly inverting the cylinder and returning it gently to its initial position. This operation requires 1 s; repeat this operation three times.

Add V_2 ml of water of known calcium hardness (8.1.3) and mix as before after sealing the cylinder. Repeat the operation five times and maintain the cylinder at the selected test temperature in the thermostatically controlled bath (7.3) for 5 min. Mix again as before, repeat the operation five times and then, using a suitable device, attach (see the figure) one of the 10 ml pipettes (7.2), closed at its upper end, to the cylinder, so that the tip is approximately 1 cm from the bottom of the graduated cylinder.

Replace the graduated cylinder in the thermostatically controlled bath for 1 h and then, using one of the 10,0 ml pipettes (7.2), take 10 ml of the solution and titrate it with the hydrochloric acid solution (6.3) in the presence of 3 drops of bromocresol green solution (6.4) to a sharp colour change from blue to green.

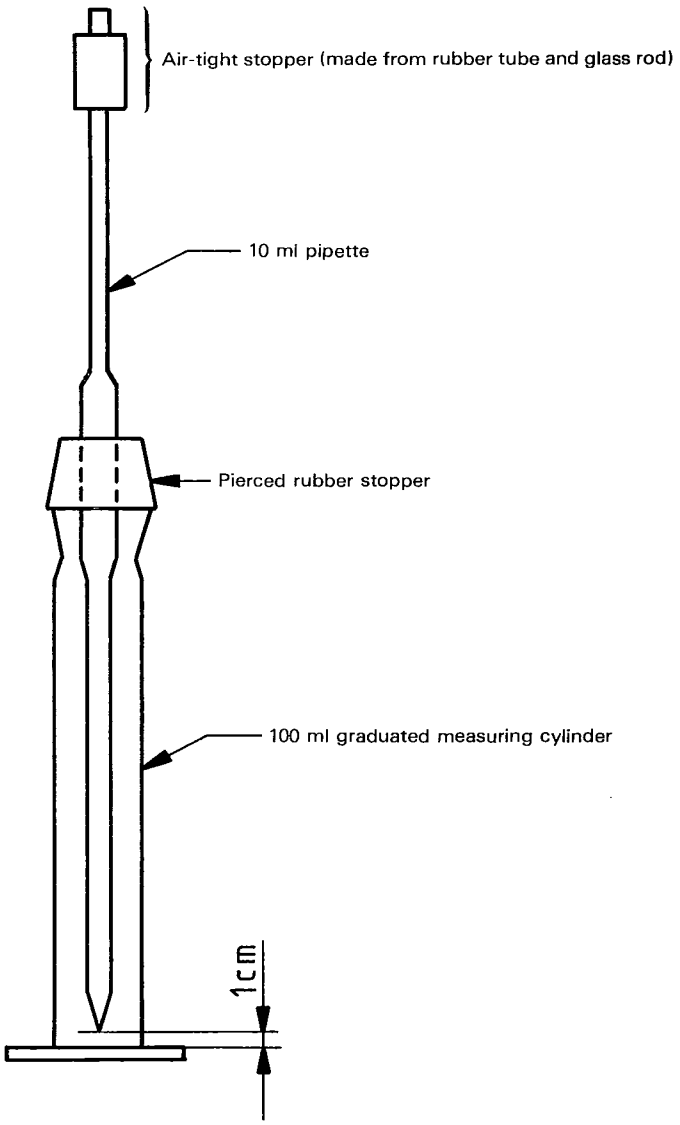
Carry out a series of tests using water of known hardness with varying additions of dispersing agent solution so that it will be possible to determine the minimum volume of dispersing agent solution, $V_{1,\,min}$, such that

$$V_3 \geqslant 0{,}95\ V_0$$

where

V_0 is the volume, in millilitres, of the hydrochloric acid solution (6.3) used for the titration of the initial soap solution (8.2);

V_3 is the volume, in millilitres, of the hydrochloric acid solution (6.3) used for the determination of the solution of calcium soap free from flocculated soap.



Figure

9 Expression of results

9.1 Method of calculation

The power to disperse calcium soap, expressed by the quantity of soap dispersed divided by the minimum quantity of dispersing agent, is given by the formulae

$\frac{100}{V_{1,\,min}}$ for a 0,1 % (m/m) solution of dispersing agent

$\frac{20}{V_{1,\,min}}$ for a 0,5 % (m/m) solution of dispersing agent

9.2 Precision

Comparative tests carried out by nine laboratories on three surface active agents at a temperature of 40 °C on a 14,28 meq/l solution of soap in hard water produced the statistical data shown in the following table.

Surface active agent	A	B	C
Mean	14,0	21,6	24,3
Standard deviation of reproducibility, σ_R	2,5	2,2	2,2

10 Test report

The test report shall contain the following information :

a) all the information required for the full identification of the sample;

b) the reference of the method used (reference to this International Standard);

c) the results obtained and the form in which they are expressed;

d) the testing conditions

1) the temperature of the thermostatically controlled bath,

2) the hardness of the water used;

e) any operational details not in this International Standard or which are optional, together with any occurrences likely to have affected the results.