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Surface active agents — Determination of surface tension

The European Standard EN 14370:2004 has the status of a British Standard

ICS 71.100.40

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June 2009

British Standards

National foreword

This British Standard is the official English language version of EN 14370:2004.

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;
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English version

Surface active agents - Determination of surface tension

Agents de surface - Détermination de la tension superficielle

Grenzflächenaktive Stoffe - Bestimmung der Oberflächenspannung

This European Standard was approved by CEN on 1 July 2004.

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Contents

Foreword

This document (EN 14370:2004) 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of an identical text or by endorsement, at the latest by March 2005, and conflicting national standards shall be withdrawn at the latest by March 2005.

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1 Scope

This document specifies test methods for the determination of surface tension of liquids, particularly surface active agent solutions.

The methods are suitable for determining the static surface tension of liquids, for example inorganic and organic liquids and solutions.

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 304, *Surface active agents – Determination of surface tension by drawing up liquid films*

3 Principle

The force, F, exerted either by the surface tension on a plate which is brought vertically into contact with the liquid and is completely wetted by it (static method) or which is necessary to pull a horizontally suspended stirrup or ring out of the liquid surface (quasi-static method) is measured (see [1] and [2]). The surface tension is obtained by calculation (see Clause 8).

In the static method, the plate is stationary so that an equilibrium value is obtained. The quasi-static method requires movement of the ring or stirrup. By using sufficiently small and slow changes in ring or stirrup position during the measurement, deviations from equilibrium are minimised.

4 Apparatus

4.1 Tensiometer

The tensiometer shall be designed for a plate, stirrup or ring, and shall consist mainly of two parts:

a) support for the sample vessel in the form of a small horizontal platform which can be moved up and down, for example using a micrometer screw;

b) apparatus for measuring the force exerted on the plate, stirrup or ring; the uncertainty of measurement shall not exceed $\pm 10^{-6}$ N, which corresponds to a maximum error of ± 0,1 mg in determining the mass.

NOTE Instead of a torsion balance, a lever balance or an electronic balance (laboratory, analytical or microbalance) can be used as an alternative. To obtain higher efficiency and reproducibility, an automatic tensiometer incorporating a balance, motor-driven platform and evaluation unit should be used.

4.2 Plate

The plate shall consist of platinum or platinum/iridium sheet. It shall be about 20 mm long, about 0,2 mm thick and at least 10 mm high, and shall be roughened to improve its wetting power.

NOTE Satisfactory roughening can be achieved by sandblasting, by rubbing with emery or by sintering a platinum black layer onto the platinum sheet.

The plate shall be suspended at a point in the axis of symmetry. The platinum, platinum/iridium plate shall have an effective height of at least 10 mm to keep wetting errors as low as possible (see Figure 1).

4.3 Stirrups

Stirrups shall be made of platinum or platinum/iridium wire. The measuring wire shall have a diameter of (0,1 ± 0,01) mm and a length of 10 mm to 40 mm; the two branches shall be at least 10 mm long.

NOTE Two small platinum balls may be attached to the lower ends of these branches as counter-weights, or, alternatively, the lower ends may be joined by a platinum wire (see Figure 2 for an example).

Dimensions in millimetres

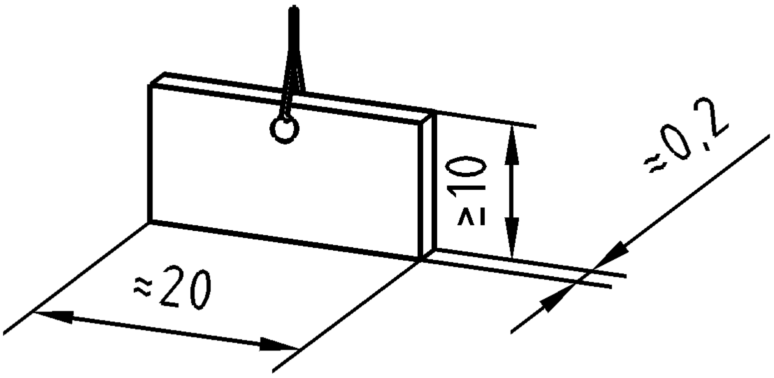


Figure 1 — Dimensions of plate

Dimensions in millimetres

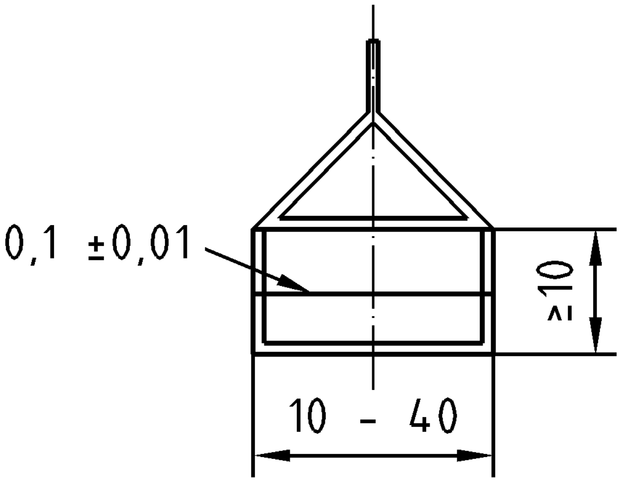
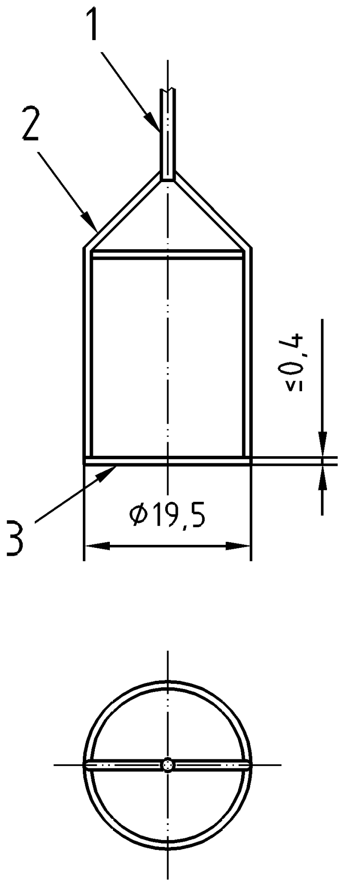


Figure 2 — Stirrup

4.4 Ring

The ring shall consist of platinum or platinum/iridium wire with a thickness not greater than 0,4 mm and a mean circumference of 60 mm (for example: inner diameter 18,7 mm, outer diameter 19,5 mm). It shall be suspended horizontally from a wire loop on a metal rod, which joins it to the tensiometer (see Figure 3 for an example).

Dimensions in millimetres



Key

1 Metal rod
2 Wire loop
3 Ring

Figure 3 — Measuring ring for the tensiometer

4.5 Sample vessel

The sample vessel for the liquid under test shall be a glass vessel, which is suitable for use in a thermostat. It shall have a sufficient capacity for the liquid, which, if necessary, shall be protected from evaporation. It shall be constructed such that the temperature of the gas phase above the liquid can also be kept constant by thermostat. There is no need to use a thermostat for measurement at room temperature. The sample vessel shall consist of a cylindrical glass container with an internal diameter of at least 45 mm, for use with the ring method, stirrup method or plate method.

NOTE If a vessel of less than 45 mm diameter needs to be used (for example, if only small amounts of sample are available), wall effects can cause an error in the surface tension measurement. The magnitude of this error depends on the distance between the wall of the vessel and the edge of the ring or stirrup.

5 Preparation of apparatus

5.1 Cleaning

Clean the glass vessels carefully, first by thorough preliminary cleaning using a suitable solvent, for example 2-propanol or propanone. Then rinse them repeatedly with freshly double distilled water and dry them.

Clean and rinse the plates, stirrups or rings in the same manner, and then heat them briefly to incandescence in a methanol or ethanol flame (the latter may be preferred since it has a low vapour pressure and higher flash point), preferably a non-luminous natural gas flame, ensuring they are only heated to red heat and never to white heat.

NOTE Should the plates, stirrups or rings become contaminated with residues, for example from silicones or cationic surface active agents, it may be preferable to clean with a sulfo-chromic acid mixture or as specified in ISO 304.

5.2 Measurement preparation

5.2.1 Principle

The zero setting of the apparatus is checked, and the tensiometer is adjusted such that results can be reliably read off from the display as millinewtons per meter.

5.2.2 Levelling

Level the apparatus by adjusting the screws at the foot of the apparatus using suitable aids, for example a spirit level placed on the tensiometer support. A protective covering against drafts may be used.

NOTE The apparatus should be mounted via rubber feet on a stone slab of about 30 kg to avoid the effect of mechanical impact and vibration (for example building vibration).

5.2.3 Zero setting

Adjust the zero point of the tensiometer, taking into account the following:

a) if a plate is used:

the zero point of the apparatus shall be adjusted after mounting the plate in the air;

b) if a stirrup is used:

the zero point of the apparatus shall be adjusted at the depth of the immersed stirrup at which the maximum force is read off. Such, no correction for the buoyancy due to the volume of the immersed connection wire or the two platinum balls is necessary (see 7.2).

NOTE This can be achieved, for example, if, when reaching the maximum force, either the lamella spread from the measuring wire is destroyed, or if, without lamella formation, the tensiometer platform height corresponding to the maximum force is set with the micrometer screw (see 4.1 a);

c) if a ring is used:

the zero point of the apparatus shall be adjusted after mounting the ring in air.

5.2.4 Calibration procedure

Calibrate the torsion balance using either of the following two methods:

a) place riders of known mass between 0,1 g and 1 g on the plate, stirrup or ring. The method is time-consuming, but the potential error is very low.

Use equation (1) to determine the apparatus factor, Φ_a, by which the measurement values shall be multiplied:

$$\phi_a = \frac{\sigma_r}{\sigma_a} \tag{1}$$

where

$$\sigma_r = \frac{m_r \times \mathrm{g}}{l} \tag{2}$$

m_r is the mass of the rider, in grams;

g is the acceleration due to gravity (981 cm/s^2);

l is the wetted periphery of the plate, stirrup or ring, that is twice the length plus twice the thickness of the plate, or twice the length of the stirrup test wire, or twice the mean circumference of the ring, in centimetres;

σ_a is the tensiometer reading after the rider is placed on the plate, stirrup or ring, in millinewtons per metre;

b) use pure substances whose surface tension is known exactly. The density of the pure calibration substance has to be the same as that of the sample fluid in order to avoid different buoyancy. This method is quicker, but less reliable.

Use equation (3) to determine the apparatus factor, ϕ_b, by which the measurement values shall be multiplied:

$$\phi_b = \frac{\sigma_o}{\sigma_g} \tag{3}$$

where

σ_o is the theoretical value of the surface tension, in millinewtons per metre;

σ_g is the measurement value of the surface tension, in millinewtons per metre.

NOTE Automatic tensiometers can be calibrated via a potentiometer setting such that subsequent conversion of the readings is not necessary.

6 Sample preparation

Use double distilled water to prepare aqueous solutions.

During measurements, keep the solution temperature constant to within ± 0,5 °C at a value which ensures a homogeneous (single phase) solution at the given concentration; micelles shall not be considered as a separate phase in this context. If there is an upper miscibility limit (for example a cloud point with aqueous ethylene oxide adducts), choose a temperature below this limit for the measurements. Always choose a measurement temperature suited to the respective problem. Measurements at room temperature are the least problematic. Thermostat the sample in the apparatus during measurement using a thermostating jacket.

NOTE 1 A difference in temperature of 1 °C causes a change of at least 0,1 mN/m; with pure water, the following relationship applies:

$$\frac{\Delta\ \sigma}{\Delta\ T} = 0{,}15\ \frac{mN}{m \times {}^\circ C} \tag{4}$$

Since the surface tension of solutions changes with time and the effect of the chemical nature of the surface active agent, its concentration and its adsorption capacity are of importance in these changes, it is not possible to specify an exact time to begin the measurement procedure. It is therefore be necessary to measure the surface tensions repeatedly as a function of time and thus of the age of the surface. After a time, which is characteristic of each system, no more changes in surface tension are observed, since equilibrium has been established.

NOTE 2 For time-dependent measurements, the static method using a plate will be the method of choice. If automatic tensiometers are used, quasi-static and static measurements can be recorded as a function of time, for example, using x/t recorders.

The surfaces of surface active agent solutions are extremely sensitive to contamination due to atmospheric dust or solvent vapours. For this reason, do not use volatile substances in the measurement room and protect the apparatus by a bell jar of the type used for balances. This will also make it easier to keep the apparatus at a constant temperature.

7 Procedure

7.1 General

Carry out several consecutive measurements under the same conditions to check the repeatability. Transfer the liquid under test to a carefully cleaned sample vessel and place the latter on the tensiometer platform.

7.2 Plate method

In the static method, bring the plate into contact with the liquid by moving the sample vessel. To avoid errors due to buoyancy, ensure that the lower edge of the plate is at the same height as the liquid surface. Note the reading on the scale; this reading is proportional to the surface tension. See [4] to [7].

For testing if the plate is wetted completely, bring the plate into contact with the liquid and read off the force. Then raise the vessel containing the test liquid by 2 mm and lower it again by the same amount, so that the lower plate boundary is back at the same height as the liquid surface. The force, then read off, shall be identical to that one measured initially. If the plate is not completely wetted, or if the meniscus of surface active agent solutions is uneven, repeat the cleaning procedure according to 5.1.

7.3 Stirrup method/ring method

Raise the sample vessel on the platform so that the stirrup or ring is below the surface of the solution under test.

If a torsion balance is used, keep the balance beam in the same position throughout the measurement by increasing the torsion of the wire as displayed on the instrument scale and simultaneously lowering the liquid level. Report the maximum force necessary to pull the stirrup or ring from the surface as the measurement value. In the case of solutions, which form films, avoid detachment if possible, and ensure that the stirrup or ring is re-immersed after the measurement is complete. This procedure reduces any disturbance in the adsorption equilibrium at the surface after measurement. See [8] to [13].

To obtain exact values for surface tension, it is necessary to measure the maximum force on pulling the stirrup or ring out of the liquid. Because of the great risk of detachment in the case of manual apparatus, extremely smooth manipulation is necessary since the value obtained immediately prior to detachment of the film is not identical to the maximum value, but will generally be some tenths of a millinewton per meter below it. Automatic tensiometers can determine the maximum electronically and reverse the platform movement promptly prior to detachment of the film. This makes it possible to obtain reliable, accurate time-consistent serial measurements without tearing the film.

Using a stirrup or ring for the measurements, the liquid surface area will be increased. If the surface active agent substance diffuses into the surface more slowly than the surface forms, this can result in errors. However, this source of error shall only be taken into account with very highly diluted surface active agent solutions and depends on the critical micelle concentration (CMC).

In addition, the kinetics of the establishment of equilibrium shall be considered. As a guide, equilibrium is established very rapidly above and in the range of the critical micelle concentration of a surface active agent. However, as dilution and molecular size increase (in particular in the case water-soluble surface active polymers) the time required to establish equilibrium increases and, in extreme cases, can amount to several hours. This means that care shall be taken to ensure that the equilibrium values are actually obtained when making measurements.

With automatic tensiometers having a built-in evaluation unit, check the establishment of equilibrium from the scattering of several (for example five) measured values. If the standard deviation falls below a small user-specified value (for example 0,1 mN/m), the measurement shall be finished. Then the static surface tension shall be the mean of the last five measurements.

8 Expression of results

8.1 General

For reasons of practicality (for example density reported in g/cm^3), perform all calculations using the centimetre, gram, second (cgs system) (for example g = 981 cm/s^2 and ring wire diameter d = 0,0370 cm). This will give the surface tension in mN/m (1 mN/m = 1 dyn/cm).

The surface tension, σ, expressed as millinewtons per metre, is given by equation (5):

$$\sigma = \frac{F}{l} = \frac{m \times \mathrm{g}}{l} \tag{5}$$

where

F is the force exerted on the plate, stirrup or ring, in millinewtons;

m is the mass of the liquid ascending, or drawn up by, the plate, stirrup or ring by capillary force, in grams;

g is the acceleration due to gravity (981 cm/s^2);

l is the wetted periphery of the plate, stirrup or ring that is twice the length plus twice the thickness of the plate, or twice the length of the stirrup test wire, or twice the mean circumference of the ring, in centimetres.

8.2 Plate method

Use equation (5) to evaluate the measurement, taking into account the apparatus factor as specified in a) or b) of 5.2.4.

8.3 Stirrup method

The value σ obtained from equation (5), taking into account the apparatus factor as specified in 5.2.4 a), is only an approximation in the stirrup measurement; it shall be corrected using the following approximation equation (6) described by Lenard [8]:

$$\sigma_L = \sigma - \frac{d}{2} \times \left(\sqrt{2\,\sigma \times \rho \times g} - \frac{2\sigma}{l} \right) + d^2 \times \frac{\pi}{16} \times \rho \times g \tag{6}$$

where

σ_L is the surface tension corrected according to Lenard [8], in millinewtons per metre;

σ is the raw value from equation (5), after applying the apparatus factor obtained as described in 5.2.4 a), in millinewtons per metre;

d is the diameter of the stirrup wire, in centimetres;

ρ is the density of the liquid under test, in grams per cubic centimetres;

g is acceleration due to gravity (981 cm/s^2);

l is the length of the stirrup measuring wire, in centimetres.

The calibration procedure as specified in 5.2.4 b) avoids the need for the Lenard approximation equation (6). In this case, multiply the raw value from equation (5) only by the apparatus factor Φ_b from equation (3).

8.4 Ring method

The value obtained from equation (5), after applying the apparatus factor obtained as specified in a) or b) of 5.2.4 is also only an approximation in the case of the ring method; correct it for the four parameters comprising the raw value of σ, the density ρ, the ring diameter D and the ring wire diameter d.

NOTE If a ring is used, equation (5) can also be expressed as follows:

$$\sigma = \frac{F}{2\,\pi\,D} \tag{7}$$

where

D is the mean diameter of the ring, in centimetres.

Equation (7) is only a rough approximation since it assumes that the surface tension acts perpendicularly on the inner and outer edge of the ring immediately prior to detachment. However, this is not the case, as the meniscus curves are not symmetrical. These effects shall be taken into account by introducing a correction factor f into equation (7).

Harkins and Jordan [10] have deduced that f is a function of D^3/V and D/d, where $V = m_\phi/\rho$ (the volume of liquid drawn up by the ring immediately prior to detachment), D is the mean ring diameter, d is the ring wire diameter, ρ is the density of the liquid and m_ϕ is the mass of the liquid drawn up. Empirically determined values of f are tabulated in [10] for a large range of D^3/V and D/d (respectively R^3/V and R/r).

Extrapolation formulae have been reported by Zuidema and Waters [11] for low D^3/V values [11] and by Fox and Chrisman [12] for high D^3/V values [12]. In automatic tensiometers having a built-in evaluation unit, a software program performs the necessary corrections, e.g. using the Zuidema and Waters method. Since it is highly laborious to determine f from the tables [10] individually for each measurement, it is convenient to take the surface tension, corrected as described in [10], directly from the tables due to Finzel and Seemann [13]. This assumes that the apparatus has been calibrated as described in 5.2.4 a), but for relative measurements, the procedure as described in 5.2.4 b) can also be used.

Corrected surface tension values of measurements made with water or dilute aqueous solutions (density, $\rho \approx 1$ g/cm^3) and rings with D = 19,100 mm and d = 0,370 mm are given as an example in Table 1. The value for σ has been obtained by multiplying the reading by ϕ_a or ϕ_b (as described in 5.2.4), while σ_k is the corrected surface tension which has been corrected by the method described in [13] in the case of the procedure specified in 5.2.4 a), or, in the case of the method specified in 5.2.4 b), has been additionally normalized to the value 72,0 mN/m at 20 °C (water).

Table 1 — Corrected surface tension values from measurements with water or dilute aqueous solutions with $\rho \approx 1$ g/cm^3 and by using rings with D = 19,100 mm and d = 0,370 mm

σ mN/m	σ_k mN/m		σ mN/m	σ_k mN/m	
	5.2.4 a)	5.2.4 b)		5.2.4 a)	5.2.4 b)
20	16,9	18,1	50	45,3	48,6
22	18,7	20,1	52	47,3	50,7
24	20,6	22,1	54	49,3	52,8
26	22,4	24,1	56	51,2	54,9
28	24,3	26,1	58	53,2	57,0
30	26,2	28,1	60	55,2	59,1
32	28,1	30,1	62	57,2	61,3
34	29,9	32,1	64	59,2	63,4
36	31,8	34,1	66	61,2	65,5
38	33,7	36,1	68	63,2	67,7
40	35,6	38,2	70	65,2	69,9
42	37,6	40,3	72	67,2	72,0
44	39,5	42,3	74	69,2	-
46	41,4	44,4	76	71,2	-
48	43,4	46,5	78	73,2	-

9 Precision

The uncertainty of surface tension measurement is considerably dependent on the nature of the liquid under test.

If the liquid is physically pure, an uncertainty of ± 0,1 mN/m can be expected using the calibration procedure specified in 5.2.4 a). For the purposes of this document, a physically pure liquid is one whose surface is free from any foreign admixtures, which can influence the surface properties.

The surface tension of solutions can alter greatly as a function of time. This is particularly marked with large molecules and concentrations greatly below the critical micelle concentration of the surface active agent. In systems of this type it will be necessary to wait for a relatively long time before carrying out measurements. Keep this time constant within a series of measurements. (See also [14] and [15].)

Small amounts of highly surface active contaminants of the substances under test can accumulate in the surface to a greater extent than the substance under test itself. This can falsify the result. Remove the top liquid layers repeatedly by suction to avoid substantially the source of error.

10 Test report

The test report shall include the following information:

a) a reference to this document;

b) the type, designation and degree of purity of the dissolved substance;

c) the type, designation and degree of purity of the solvent;

d) the concentration of the solution;

e) the test temperature, in degrees Celsius;

f) the age of the surface at the time of measurement;

g) the period for establishment of equilibrium prior to measurement;

h) the corrections used;

i) the statement of whether measurements were performed using a plate, stirrup or ring;

j) the surface tension as individual values and the arithmetic mean;

k) where appropriate, the mean of at least five measurements and the standard deviation;

l) any deviations from this document;

m) the date of the test.

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