SIST

SLOVENSKI STANDARD
SIST EN 14370:2005

01-april-2005

Površinsko aktivne snovi - Ugotavljanje površinske napetosti

Surface active agents - Determination of surface tension

Grenzflächenaktive Stoffe - Bestimmung der Oberflächenspannung

Agents de surface - Détermination de la tension superficielle

Ta slovenski standard je istoveten z: **EN 14370:2004**

ICS:

71.100.40 Površinsko aktivna sredstva Surface active agents

SIST EN 14370:2005 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14370

September 2004

ICS 71.100.40

English version

Surface active agents - Determination of surface tension

Agents de surface - Détermination de la tension superficielle

Grenzflächenaktive Stoffe - Bestimmung der Oberflächenspannung

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EUROPÄISCHES KOMITEE FÜR NORMUNG

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Ref. No. EN 14370:2004: E

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of an identical text or by endorsement, at the latest by March 2005, and conflicting national standards shall be withdrawn at the latest by March 2005.

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.

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 ± 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

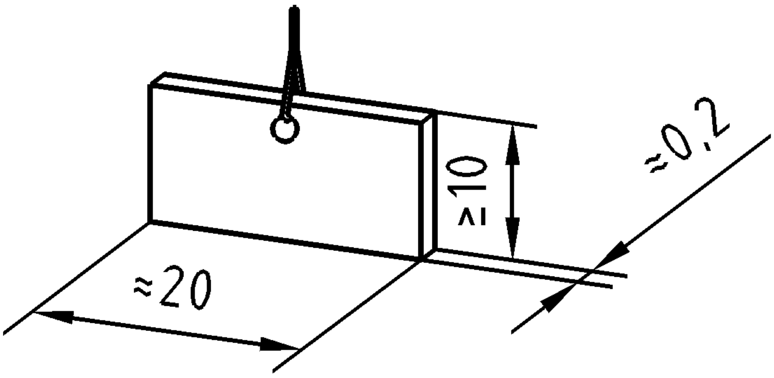


Figure 1 — Dimensions of plate

Dimensions in millimetres

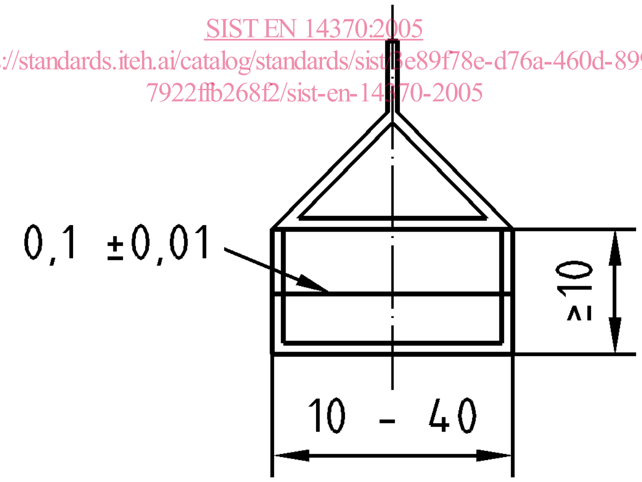
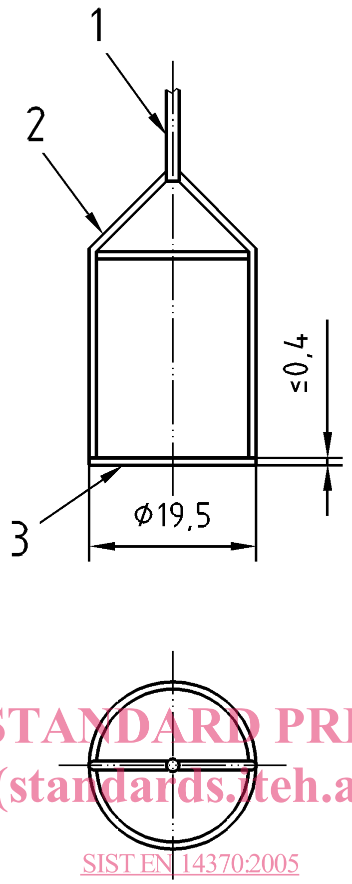


Figure 2 — Stirrup

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:

$$\phi_a = \frac{\sigma_r}{\sigma_a} \quad (1)$$

where

$$\sigma_r = \frac{m_r \times g}{l} \quad (2)$$

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:

$$\phi_b = \frac{\sigma_o}{\sigma_g} \quad (3)$$

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