
International Standard



304

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Surface active agents — Determination of surface tension by drawing up liquid films

Agents de surface — Détermination de la tension superficielle par étirement de films liquides

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 304 was prepared by Technical Committee ISO/TC 91, *Surface active agents*.

This second edition cancels and replaces the first edition (ISO 304-1978), of which it constitutes a minor revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Surface active agents — Determination of surface tension by drawing up liquid films

0 Introduction

Surface tension is a fundamental property of liquids in general and aqueous solutions of surface active agents in particular.

The measurement of this characteristic, however, does not allow, in the case of solutions of surface active agents, any suppositions as to their detergency, wetting, foaming, emulsifying activities, etc. Indeed, no connection can be established between the performance properties of the surface active agents and the surface tension of their solutions.

1 Scope and field of application

This International Standard specifies a test method for the determination, by drawing up liquid films, of the surface tension of aqueous or organic solutions of surface active agents and mixtures containing one or more surface active agents.

It also applies to the measurement of surface tension of pure liquids or solutions other than those mentioned above.

NOTE — Numerous methods have been established to determine surface tension, such as :

- a) methods by drawing up liquid films by a plate, stirrup or ring;
- b) methods for measuring the rise of liquid in a capillary tube;
- c) maximum ball pressure method;
- d) drop weight method;
- e) drop or sessile ball method;
- f) suspended drop method;
- g) dynamic methods based on measurements of flow characteristics;
- h) methods which study the waves produced in capillary jets.

The methods based on drawing up liquid films have notable advantages in the simplicity of their procedure and the commercial availability of highly automatic appliances.

2 References

ISO 862, *Surface active agents — Vocabulary*.

ISO 2456, *Surface active agents — Water used as a solvent for tests*. *

3 Definition

surface tension : See ISO 862.

NOTE — The SI unit of surface tension is the newton per metre (N/m). In practice, the submultiple millinewton per metre (mN/m)** is used.

4 Principle

Measurement of the maximum force which it is necessary to exert vertically on a stirrup or a ring, in contact with the surface of the liquid being examined placed in a measuring cup, in order to separate it from this surface, or on a plate, with an edge in contact with the surface, in order to draw up the film that has formed.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Tensiometer, which can be fitted with plates, stirrups and rings, and which shall comprise the following items :

- a) A **horizontal platform**, which can be moved vertically in both directions by means of a micrometer screw. It shall be fitted with a millimetric scale allowing estimation of vertical movements of 0,1 mm;

* At present at the stage of draft.

** 1 mN/m = 1 dyn/cm

b) A **dynamometer**, to give a continuous measure of the force applied to the measuring unit with an accuracy of at least 0,1 mN/m.

c) A **device** for indicating or recording the values measured by the dynamometer.

The assembly shall be shielded from vibrations and draughts.

5.2 Measuring unit, which shall comprise at least one of the items a), b) and c) and also item d).

a) A **rectangular plate** of sheet platinum, 0,1 mm thick and about 30 mm long by 20 mm wide, roughened using emery paper in such a way that the strokes are perpendicular to the side intended to be submerged. The plate is fixed, preferably by welding but alternatively by any other means, to a suspension rod situated in the axis of symmetry of the plate (see figure 1).

b) A **stirrup** of platinum — iridium wire of diameter not exceeding 0,1 mm; the length of the horizontal arm is 20 to 40 mm and the length of the two vertical branches is 10 mm. The ends of these branches terminate in two small platinum balls which act as counterweights (see figure 2).

c) A **ring** of platinum — iridium wire, 0,3 mm in diameter. The circumference of the ring is generally between 40 and 60 mm. It is fixed to a suspending rod by means of a platinum wire stirrup (see figure 3).

d) A **measuring cup**, to hold the test portion of the liquid to be examined, consisting of a small glass tank capable of containing a sufficient quantity of liquid. Watch-glasses shall not be used.

In the case of the determination of the surface tension of solutions of surface active agents, cylindrical tanks with a diameter of at least 8 cm may be used.

For determinations on pure liquids, the ideal measuring cup is a small tank in the form of a rectangular parallelepiped having sides of at least 8 cm; this shape enables the liquid surface to be cleaned completely by wiping.

NOTE — Generally, in the case of dilute solutions of surface active agents, the surface should only be wiped if the concentration of the surface active agent is close to the critical concentration for micelle formation (c.m.c.).

6 Procedure

6.1 Preparation of solutions of surface active agents

6.1.1 The solutions of surface active agents for the determinations shall be prepared with all necessary care. The water used for preparing them shall be double-distilled water (see sub-clause 5.2 of ISO 2456), checked by measuring its surface tension. Cork stoppers and, more particularly, rubber stoppers, shall on no account be used, either in the construction of the distillation apparatus or for stoppering containers in which the water is kept.

6.1.2 The temperature of the solutions shall be maintained to within 0,5 °C.

NOTE — Determinations carried out in the region of points of critical solubility, such as the Krafft temperature, cloud temperature of ethylene oxide condensates, etc., are seriously marred by errors. It is preferable to proceed at a temperature higher than that of the particular points, or at a temperature lower than the cloud temperature of ethylene oxide condensates.

6.1.3 As the surface tension of solutions varies with time, it is difficult to recommend a standard ageing period for the solution, because the nature and purity of the surface active agent, its concentration and its liability to adsorption all play a particular part in these variations. It is therefore desirable to make several measurements over a period of time, to construct the surface tension curve as a function of time and to determine the position of the level portion, which gives the age at which the solution has reached its equilibrium state. Automatic apparatus is very suitable for carrying out the measurements, the values being recorded as a function of the time.

6.1.4 The surface of the solutions is extremely sensitive to contamination by atmospheric dust or by vapours from solvents handled in the vicinity. Therefore no volatile products shall be handled in the room where the determinations are made and the complete apparatus shall be protected by a bell of the kind used for balances. These precautions also reduce variation in temperature.

6.1.5 The method recommended for taking the test portion of the liquid examined is to suck it, with a pipette, from the centre of the mass of liquid, as the surface may be liable to soiling by insoluble particles and dust.

6.2 Cleaning the measuring unit

6.2.1 Cleaning the measuring cup

In the presence of impurities such as silicones, which are not removed by sulfo-chromic acid mixture, phosphoric acid or a solution of potassium persulfate in sulfuric acid, wash the measuring cup using special products (for example toluene, perchloroethylene or methanolic potassium hydroxide solution).

If these impurities are absent, or after cleaning with these products, wash the measuring cup carefully with hot sulfo-chromic acid mixture and then with concentrated phosphoric acid [83 to 98 % (m/m)]. Finally, rinse in double-distilled water until the rinsings are neutral. The double-distilled water shall be freshly prepared according to sub-clause 5.2 of ISO 2456.

Before the determination, the measuring cup shall be rinsed several times with the liquid to be examined.

6.2.2 Cleaning the plate, stirrup or ring

If necessary, clean the platinum plate, stirrup or ring (5.2) with special products as specified in 6.2.1. In the absence of impurities requiring cleaning with these products, or after cleaning with these products, wash the platinum measuring unit