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# Standard Test Methods for Surface and Interfacial Tension of Solutions of Surface- ~~Active Agents~~ Paints, Solvents, Solutions of Surface-Active Agents, and Related Materials<sup>1</sup>

This standard is issued under the fixed designation D1331; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

## 1. Scope

1.1 These test methods cover the determination of surface tension and interfacial tension of a variety of liquid materials, including but not restricted to paints, solvents, and solutions of surface-active agents, as defined in Terminology D459. ~~Two~~Four methods are covered as follows:

Method A—~~Surface Tension~~: Tension by du Noüy ring.

Method B—~~Interfacial Tension~~: Tension by du Noüy ring.

Method C—Surface Tension by Wilhelmy plate.

Method D—Interfacial Tension by Wilhelmy plate.

1.2 Method A ~~is originally~~ was written primarily to cover aqueous solutions of surface-active agents, but is also applicable to aqueous paints, nonaqueous solutions (including paints) and mixed solvent solutions.

1.3 Method B is applicable to two-phase solutions. More than one solute component may be present, including solute components that are not in themselves surface-active.

1.4 Method C is applicable to surface active liquids and, unlike du Noüy ring, no buoyancy corrections are needed and results are not affected by moderate viscosities (1-10 Pa-sec) of the liquid. It is the recommended method for use with paints and resin solutions.

1.5 Method D is applicable to two-phase solutions and mixtures.

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Material Safety Data Sheets are available for reagents and materials. Review them for hazards prior to usage.

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D459 Terminology Relating to Soaps and Other Detergents

## 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *du Noüy ring*—platinum wire ring.

3.1.2 *Wilhelmy plate*—flat, thin plate made of glass or platinum.

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.24 on Physical Properties of Liquid Paints and Paint Materials.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's standard's Document Summary page on the ASTM website.

#### **4. Summary of Test Methods**

4.1 *du Noüy Ring Methods*—The ring is suspended from a tensiometer or balance. The test liquid is brought into contact with the ring until the latter is covered. For interfacial tension measurements, the ring must be at the interface between the two liquids. The ring is then slowly withdrawn until a maximum force is attained, from which the surface or interfacial tension may be calculated.

4.2 *Wilhelmy Plate Methods*—The plate is suspended from a tensiometer or balance. The test liquid or interface between two liquids is brought into contact with the bottom of the plate, causing the plate to be pulled down into the liquid by the surface tension force. The force applied to the plate from above is then increased to bring the bottom edge of the plate level with the flat surface of the liquid. The force acting on the plate is measured and used to calculate the surface tension of the liquid or the interfacial tension between two liquid phases.

#### **5. Significance and Use**

5.1 These methods provide data that are useful in evaluating the effectiveness of surface active agents in reducing surface tension. In addition, surface tension data can predict interactions between liquids and solid surfaces or other liquids and can be used to establish wetting properties of paints, solvents, and other liquids.

5.2 A number of laboratories have found the Wilhelmy plate to be easier to use, easier to clean and generally better for use with pigmented paints.

#### **6. Interferences**

6.1 The du Noüy ring is difficult to clean when used with pigmented paints. Paints high in titanium dioxide have a tendency to coat the ring with a layer that is almost impossible to remove. If left on the ring, this layer of material affects the surface tension values that are measured.

6.2 Paint measurements with the du Noüy ring are affected by viscosity above a level of 10 Poise (1 Pa-sec). Many coatings have a viscosity at rest greater than that level. Above 10 P, the resistance of the ring to being pulled is as much due to viscosity as to surface tension.

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**METHOD A—SURFACE TENSION BY DU NOÜY RING**  
 Document Preview

#### **7. Apparatus**

7.1 *du Noüy Tensiometer*—Either the du Noüy precision tensiometer or the du Noüy interfacial tensiometer, equipped with either the 4 or the 6-cm circumference platinum ring, as furnished by the manufacturer, may be used. The tensiometer shall be placed on a sturdy support that is free from vibrations and other disturbances such as wind, sunlight, and heat. The wire of the ring shall be in one plane, free of bends or irregularities, and circular. When set in the instrument, the plane of the ring shall be horizontal, that is, parallel to the surface plane of the liquid being tested.

7.2 *Sample Container*—The vessel for holding the liquid shall be not less than 6 cm in diameter, and sufficiently large to ensure that the contact angle between the ring and the interface is zero.

#### **8. Preparation of Apparatus**

8.1 Clean all glassware thoroughly. The use of fresh chromic-sulfuric acid cleaning mixture, followed by a thorough rinsing in distilled water, is recommended.

8.2 Clean the platinum ring by rinsing thoroughly in a suitable solvent and in distilled water, before taking a set of measurements. Allow the ring to dry, and then heat to white heat in the oxidizing portion of a gas flame.

#### **9. Calibration of Apparatus**

9.1 The tensiometer is, in fact, a torsion balance, and the absolute accuracy depends on the length of the torsion arm, which is adjustable. Torsion may be applied to the wire by means of either the dial-adjusting screw (which controls the dial reading) or a rear adjusting screw. Calibration consists essentially in adjusting the length of the torsion arm so that the dial scale will read directly in dynes per centimetre. The precision tensiometer shall be calibrated in accordance with the following: Calibrate the tensiometer or balance according to the manufacturer's directions. 5.1.1—5.1.3; the interfacial tensiometer shall be calibrated in accordance with 5.1.1—5.1.4.

5.1.1 Level the tensiometer. A liquid level of the type employed on analytical balances may be used. Place the level on the table that holds the sample for testing, and adjust the leg screws of the tensiometer until the table is horizontal. Pull the torsion wire taut by means of the tension screw, and adjust the dial reading and the vernier to zero. Insert the platinum ring in the holder, and place a small piece of paper across the ring. This will serve as a platform to hold the calibrating weight. Turn the rear adjusting screw of the torsion wire until the index level of the arm is opposite the reference line of the mirror; this automatically compensates

for the weight of the paper platform. Next, place an accurately standardized weight of between 500 and 800 mg on the paper platform and turn the dial-adjusting screw until the index level of the arm is opposite the reference line of the mirror. Record the dial reading to 0.10 division. Call this “gamma-c.”

5.1.2 Calculate what the reading “gamma-c” obtained in 5.1.1 *should be* when the tensiometer is properly adjusted, as follows (Note 1):

$$\gamma_c = (M \times g) / 2L \quad (1)$$

where:

- $M$  = weight placed on the paper platform, g;
- $g$  = gravity constant (Note 2), cgs units, and
- $L$  = mean circumference of the ring (furnished by the manufacturer with each ring).

If the recorded dial reading “gamma-c” is greater than the calculated value, the torsion arm should be shortened. If “gamma-c” is less than the calculated value, the torsion arm should be lengthened. Repeat the calibration procedure, readjusting the zero position after each change in the length of the torsion arm, until the dial reading agrees with the calculated value. Each unit of the scale now represents a pull on the ring of 1 dyne/cm. Note that a conversion factor,  $F$  (see 5.1.3), must be multiplied by the scale reading to give corrected surface tension in dynes per centimetre.

NOTE 1—Example—If  $M$  is exactly 0.600 g and  $L$  is 4.00 cm:

$$\gamma_c = (0.600 \times 980.3) / (2 \times 4.00) = 73.52 \text{ dynes/cm}$$

NOTE 2—The gravity constant is 980.3 at Chicago; in other localities it will differ very slightly from this value.

5.1.3 After the tensiometer has been calibrated, it is convenient to calculate the number of grams total pull on the ring that is represented by each scale division. This is done simply by dividing the scale reading into the weight used for calibration (Note 3). This value is used in the calculation of the conversion factor,  $F$ , mentioned in 5.1.2.

NOTE 3—In the example given in Note 1, each scale unit after calibration represents:

$$0.600 / 73.52 = 0.008161 \text{ g} \quad (2)$$

5.1.4 *Interfacial Tensiometer*—With the interfacial tensiometer, the same principle of calibrating by adjusting the length of the torsion arm also applies. This instrument has, however, in addition to the torsion arm, a torsion-arm counterbalance. Adjust the length of this counterbalance to coincide with that of the torsion arm itself, in order that the vertical members of the assembly may remain in line.

## METHOD A—SURFACE TENSION

### 10. Procedure

10.1 After the tensiometer has been calibrated, check the level and insert the cleaned platinum ring (Note 41) that will be used in the measurement. Check the plane of the ring, and set the dial and vernier at zero. Adjust the rear adjusting screw so that the index level of the arm is opposite the reference mark on the mirror, that is, the ring system is at the zero position.

NOTE 1—Extreme care must be taken to have the sample vessel and platinum ring clean. Contamination of the liquid surface by dust or other atmospheric impurities during measurement should be avoided.

10.2 Place the solution to be tested (Note 52), contained in the thoroughly cleaned vessel (Note 41), on the sample platform. Raise the sample platform by means of its adjusting screw until the ring is just submerged.

NOTE 2—~~Since~~ For surface active agents: since the surface tension of a solution is a function of the concentration, care must be taken that the concentration is adjusted and recorded within known limits. The presence of solutes other than the surface-active agent should be ascertained and reported qualitatively and quantitatively, insofar as possible. This includes hardness components in the water. Care should be taken that the solution is physically homogeneous. Measurements made near or above the cloud point or other critical solubility points can be in serious error. This is particularly true when the solute is a surface-active material.

10.3 Lower the platform slowly, at the same time applying torsion to the wire by means of the dial-adjusting screw. These simultaneous adjustments must be carefully proportioned so that the ring system remains constantly in its zero position. As the breaking point is approached, the adjustments must be made more carefully and more slowly. Record the dial reading-force reading from the dial or the digital display when the ring detaches from the surface.

10.4 Make at least two measurements. Additional measurements shall be made if indicated by the over-all variation obtained, the total number of readings to be determined by the magnitude of that variation.

10.5 Record the temperature of the solution and the age of the surface at the time of testing. Since the submerging of the ring (6.210.2) may constitute a significant disturbance of the surface, take the age as the elapsed time between submersion and breakaway of the ring. The accuracy of this time observation may be indicated in the usual manner. In most cases an accuracy of  $\pm 5$  s is reasonable, and sufficient for this test method.