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The Surface Tension of Synthetic Blood used for ASTM F1670 Penetration Tests

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Abstract

The American Society for Testing and Material (ASTM International) F1670 test method was based on research involving transmission of bloodborne pathogens (Hepatitis B, Hepatitis C, and HIV) in the 1980s. The test method details the measurement of synthetic blood penetration through garments. A key parameter affecting penetration is synthetic blood surface tension which is measured via du Noüy ring tensiometer. However, little is known about the sources of variation impacting surface tension measurements. In this study, the synthetic blood used for ASTM F1670 was evaluated from within the ASTM F903 test apparatus and with two mixing treatments. Measurements were compared against two outside laboratories and with two alternate tensiometric methods (pendant drop and capillary rise).

It was found that using the methods specified in the ASTM F1670 test method, surface tension of the synthetic blood was not 40–44 dynes/cm as was expected. The surface tension was initially above 50 dynes/cm and declined to below 40 dynes/cm after 60 minutes. The surface tension within the penetration cell was relatively constant over time, showing that the surface tension measurements outside the penetration cell are not indicative of the surface tension within the apparatus during the test. Shaking the synthetic blood, a mixing procedure detailed in the ASTM F1670 test method, increased the surface tension. The increase was greatest in a container having more airspace. Du Noüy ring measurements by NIOSH compared to external labs were within 15%. Testing with alternate methods showed that the “open-to-atmosphere” methods (ring and drop) began lower and declined rapidly when compared to the “closed-to-atmosphere” method (capillary). Results of this research will help amend the ASTM F1670 standard to better characterize the measurement and handling of synthetic blood used in the ASTM F1670 test and to provide a framework for consideration of test fluid used in future ASTM standards.

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Disclaimers

The findings and conclusions in this report are those of the authors and do not necessarily represent the views of the National Institute for Occupational Safety and Health. Mention of commercial product or trade name does not constitute endorsement by the National Institute for Occupational Safety and Health.

Keywords

surface tension; synthetic blood; F1670; F1862; fluid penetration; liquid penetration; strike-through; protective clothing

Background

ASTM F1670 (formerly ASTM ES-21 and 22) is a standard method that tests the resistance of synthetic blood penetration through protective clothing material. It was based on research motivated by Hepatitis B, Hepatitis C, and HIV epidemics in the 1980s [1–3]. ASTM F1670 is used to identify a garment's resistance to synthetic blood penetration and to screen for garments that are candidates for viral testing [4].

ASTM F1670 specifies the synthetic blood surface tension to be 40–44 dynes/cm. A fluid outside this range should not be used [4]. Fluid surface tension is important because fluids with a high surface tension resist penetration through fabric having small pores [1, 5].

ASTM F1670 specifies the du Noüy ring (dNR) method [6]. The dNR method utilizes the interaction of a platinum ring with the surface of the liquid [7]. The ring is submerged below the interface by moving the stage where a liquid container is placed. After immersion, the stage is gradually lowered and the ring pulls up a meniscus from the liquid. Surface tension is determined at the instant the meniscus tears from the ring. The ring method is accurate for pure fluids, such as water, and reasonably accurate for fluid mixtures that reach equilibrium within a few seconds [8–10]. The measurement of surface tension is time dependent, with dynamic (changing) and static (stable) phases (Fig. 1). During the dynamic phase, surfactant molecules are drawn by convection to the surface due to "stretching" of the fluid. The surface tension is maximum when stretching begins and comparable to a liquid without impurities. During the dynamic phase, the surface tension of the fluid decreases. Once the molecular forces between a surfactant in a bulk fluid and its surface reaches equilibrium, the static phase is established. This appears as a plateau (i.e., asymptote) in the curve, and is also known as the equilibrium time [8].

There are several methods for measuring the surface tension of fluids. Some measure the dynamic phase while other methods measure the static phase. Some methods are more accurate, some are more time consuming, and some work better with certain fluid types. Methods also differ in the amount of exposure to external conditions. The pendant drop method is open to the atmosphere on all sides, the du Noüy method is open to the atmosphere on one side, and the capillary rise method is nearly entirely closed to the atmosphere (Fig. 2).

The pendant drop method derives surface tension by the shape of a drop hanging from a needle. The shape is a function of a balance of forces between gravitational pull against the liquid, liquid density, and the cohesion within the drop [11]. The capillary rise method is the oldest method used to determine surface tension [12]. The height of fluid within a capillary is taken at the time when the interaction forces of the liquid with the capillary walls (adhesion) are stronger than those between the liquid molecule's cohesion. Initially,

the liquid wets the walls and rises in the capillary tube. Mechanical equilibrium is attained when the pressure equalizes between the liquid and the capillary wall surface.

The objectives of this study were: 1) to show that the surface tension of the synthetic blood is not 42 dynes/cm, as the F1670 standard claimed for the past three decades; and 2) to describe the surface tension with respect to time, location, measurement technique, and handling. As characterized by Defay [8], we expect the surface tension measured by dNR will decline over time. Since the penetration cell is a closed atmosphere, we expect the surface tension within the cell will not decline during the one hour F1670 test. Also, since bubbles were observed on the fluid surface after shaking the synthetic blood, we expect that excessive shaking will affect the surface tension.

Materials and Methods

Synthetic blood composed of Acrysol g-111, mineral oil, direct red 81 dye, and HPLC water was purchased in one liter bottles and was stored at room temperature (22±2 °C) (Johnson, Moen & Co. Inc., Cannon Falls, MN). Surface tension was measured with a du Noüy Precision Tensiometer (Model 70535, CSC Scientific Company, Inc., Fairfax, VA) in a constant climate chamber (model HPP 260, Memmert, Germany), which was monitored and recorded with a temperature/humidity meter (Model CNiTH-i8DH33–2, OMEGA Engineering, Inc, Stamford, CT).

To mix and extract with minimal infusion of dissolved air, a technique of inverting and swirling the synthetic blood was developed, herein referred to as “mixed and extracted.” The container was inverted, gently swirled five times, and reinverted. Fluid was drawn into a new syringe from a middle depth of the container; never from the surface or the bottom. Near empty containers were discarded to prevent the possibility of analysis of surface oils, surface air, or sediment particulates. Each syringe was first primed with one milliliter of synthetic blood that was dispensed before a quantity of test fluid was drawn. The syringe plunger was moved slowly to minimize depressurization.

To correspond with the ASTM F1670 test procedure which lasts 60 minutes, dNR measurements began immediately after dispensing the fluid into a petri dish, which took approximately one minute for the first reading, followed by readings at 5, 10, 20, 40, and 60 minutes. Between readings, the ring remained submerged three millimeters below the surface. After each 60 minute test, the ring was rinsed in ultrapure distilled water and cleaned by flame (ethanol 95% pure).

MEASURED FROM PENETRATION CELL

The penetration cell was filled with 60 milliliters of synthetic blood. Seven milliliters were drawn from the center of the penetration cell and dispensed into a 47 millimeter petri dish. Each sample was measured five times in succession using the dNR method.

Because the surface tension of the synthetic blood constantly changes while in an undisturbed petri dish, repeat measurements are not possible. However, these five successive measurements capture a rate of decline.

This procedure was repeated with synthetic blood remaining in the penetration cell for 0, 5, 10, 20, 40 and 60 minutes. Control samples were taken directly from the fluid container for a total of six samples and two controls. Measurements were conducted at 50±5% relative humidity and at 22±2 °C.

INTER-LAB COMPARISONS

The dNR measurements were confirmed with two external laboratories (Medline Industries, Inc. and FDA's Center for Devices and Radiological Health). In these comparisons the 47 mm petri dish was replaced with a 100 mm × 15 mm, sterile, polystyrene petri dish (cat# FB0875712, FisherScientific, Hampton, NH) to conform with the Standard Test Methods for Surface and Interfacial Tension of Solutions of Paints, Solvents, Solutions of Surface-Active Agents, and Related Materials [6] that specifies use of a dish having a diameter greater than six centimeters (The 100 mm × 15 mm polystyrene petri dish has an actual internal diameter that measures 87 mm and will be referred to as an 87 mm petri dish).

Dial readings on the du Noüy tensiometer were multiplied by a ring F factor reported by each laboratory for each measurement. Temperature and humidity conditions were reported by each laboratory. The same lot of synthetic blood was shared between all laboratories. For the Medline comparison synthetic blood was "mixed and extracted," and 20 ml was slowly dispensed into a new 87 mm petri dish. Each measurement series was repeated three times.

The FDA comparison experiment was conducted from the same synthetic blood lot, but four months after the Medline comparison. The test procedure was slightly modified to achieve closer agreement. The edge of the petri dish that holds the test fluid also affects the intermolecular attractions, changing the stability of the measurement and causing wall-compression effects [13]. Rather than dispense 20 ml of synthetic blood into each petri dish, a greater volume (35 ml) of synthetic blood was dispensed into the 87 mm petri dish. Also, for the FDA comparison both labs shared the same wire ring. Each measurement series was repeated five times.

PENDANT DROP AND CAPILLARY RISE MEASUREMENTS

The pendant drop test was performed with a Theta Lite TL101 configured with OneAttension version 2.5 (Biolin Scientific, Espoo, Finland). The pendent drop Young-Laplace analysis mode was chosen with a synthetic blood fluid density of 1.00. The synthetic blood fluid density was validated using a ML204 microbalance (Mettler Toledo, Greifensee, Switzerland) and a 100 ml volumetric flask.

Capillary Rise tests were performed with a 0.5 mm graduated capillary tube (Cat# 14–818, FisherScientific, Hampton, NH). The actual capillary diameter was calibrated with Benzene as specified in the instruction manual.

SHAKING CONTAINERS WITH DIFFERENT FLUID AMOUNTS

The synthetic blood was "mixed and extracted" into 30 mm × 115 mm style conical tube (Bectofi-Dickinson 352070 BlueMax 50mL Polypropylene, Nonpyrogenicsci). Tubes (58 ml actual volume) were filled with either 10 ml or 50 ml of synthetic blood. For this experiment

the pendant drop method was chosen over the dNR method because it requires only a few drops of test fluid allowing measurements each hour for several hours without depleting the volume of test fluid. An initial measurement was obtained before shaking. Tubes were shaken vigorously for one minute. Each measurement series was repeated two times.

Statistics

Statistical differences are evident with cursory statistical analysis. The averages, standard error, and trendlines were calculated in Microsoft Excel (2013).

Results

MEASURED FROM PENETRATION CELL

Each dNR measurement lasted between one and two minutes. All five dNR measurements were completed within nine minutes for all eight samples. The decline of surface tension is within one dyne/cm regardless of the time of extraction from the penetration cell or if the fluid was extracted from the fluid vessel (Fig. 3).

INTER-LAB COMPARISONS

Synthetic blood measurements were compared between NPPTL's lab and two outside labs (Fig. 4 and Fig. 5). The results were within 15% compared to Medline (40±10% RH) and within 8% compared to FDA (50±10% RH). All labs confirmed that the first surface tension measurement of the ASTM F1670 synthetic blood began near 50 dynes/cm. Over time, the surface tension decreased for all experiments. For all three labs, at 20 minutes the surface tension agreed with the value specified in the ASTM F1670 standard (40–44 dynes/cm).

PENDANT DROP AND CAPILLARY RISE MEASUREMENTS

For the pendant drop method, the density of synthetic blood at 21°C was 100.34 g per 100 ml (1.003 g/ml). For the capillary rise method, the actual diameter of the capillary tube was calculated to be 0.487 mm according to the following calculation. The National Institute of Standards and Technology (NIST) tables list the surface tension of benzene at 21°C to be 28.73 dynes/cm and the density to be 0.8778 g/cm^3. A gravity constant of 980.7 cm/sec^2 was used for latitude 40.305, longitude −79.980. Capillary height of benzene was measured five times; the average value was 2.74 cm.

The surface tension of both "open" methods decreased rapidly over time while the capillary method decline remained relatively flat (Fig. 6). The pendant drop method, being exposed to the atmosphere on all sides, had the fastest decline in surface tension (from 55 dynes/cm to 43 dynes/cm in 11 minutes; shortly after this time the drop becomes brittle and falls from the dispensing needle). The dNR method exposed to the atmosphere on only one side declined nearly as fast as the pendant drop (from 52 dynes/cm to 40 dynes/cm after 20 minutes). The capillary method (closed atmosphere) remained nearly constant (from 61 dynes/cm to 60 dynes/cm after 60 minutes). Surface area to volume ratios (SA/V) pertaining to fluid testing are summarized (Table I).

SHAKING CONTAINERS WITH DIFFERENT FLUID AMOUNTS

Synthetic blood measurements were compared after shaking bottles with different amounts of fluid and airspace in containers. One factor of time pertains to the amount of time the drop was exposed to external atmosphere. Another factor of time was how many hours passed since the synthetic blood was shaken. To show the rise and subsidence of surface tension in each passing hour, we compare drops having the same exposure to atmosphere (500 seconds) (Fig. 7). Variability in the data was greatest with the first measurement where the repeated sample differed by 2.5 dynes/cm. A log fit trendline indicates that shaking increased the surface tension in both samples. The bottle with greater air space increased more and also subsided faster towards the pre-shake value.

Summary and Conclusions

The surface tension of the ASTM F1670 synthetic blood was measured under a range of modified experimental conditions. Effects of aeration from shaking the fluid to mix its components, and testing directly from the penetration cell apparatus were evaluated. Measurements by du Noüy method were compared between two other labs and two alternate methods were considered (pendant drop and capillary rise).

Results confirmed that shaking the synthetic blood fluid changes the dNR surface tension measurement. One possible explanation is that residual dissolved air can enhance intermolecular partial hydrogen bond attractions [14]. Because fluids with low surface tension are more penetrable, further experimentation is needed to determine if ASTM F1670 tests results are affected by fluid aeration. An alternative is not to aerate the fluid, but rather gently mix the fluid with a procedure that does not change the surface tension measurement. Results confirmed that surface tension of the synthetic blood is relatively stable within the cell's "closed-to-atmosphere" environment.

The dNR surface tension measurements between labs were all within 15%. Many factors can potentially cause variable results [e.g. the instrument (model and calibration); the ring (condition, specification and cleanliness); the ring F correction factor; the fluid mixing and dispensing technique; environmental conditions (temperature, humidity, elevation/gravity, and atmospheric pressure); the dish (material, size, and fluid volume); the operator's technique; and the time of measurement]. Some fluids, such as synthetic blood, are highly sensitive to many of these factors, which is perhaps why the surface tension was not previously characterized.

To determine the surface tension of synthetic blood, three methods were compared (pendant drop, dNR, and capillary rise). One limitation of this study is that all three methods provide only static measurements and are not capable of dynamically measuring surface tension of fluid mixtures that change over time. The three methods do provide different amounts of exposure to the outside atmosphere, however, without knowing the equilibrium time, the accuracy of the measurements cannot be determined. For a mixture with a solute (surfactant in this case) that lowers the surface tension of a pure fluid, any measurement prior to equilibrium being reached would be high. In non-saturated atmospheres above the fluids surface, measurements made with a relatively long equilibrium time would begin

to evaporate, effectively changing the solution's concentration, resulting in a confounding effect by most likely lowering its actual surface tension irrespective of the equilibrium time. Thus, a problem leading to inaccuracies of both the dNR and pendant drop methods are that the fluid is exposed to the atmosphere. In contrast, the capillary rise method, which is the oldest method used for surface tension determination [12], is closed to the atmosphere and is unaffected by ambient climate. It is the complex and changing nature of the synthetic blood fluid that makes characterization of the fluid and inter-lab comparisons difficult.

As validated by three laboratories, the surface tension of the synthetic blood was not 40–44 dynes/cm. We conclude that the surface tension of synthetic blood used in ASTM F1670 depends on the method and time of measurement. Shaking the synthetic blood, as specified by the ASTM F1670 test method, affects the surface tension. The surface tension of synthetic blood remains stable in the 'closed to atmosphere' conditions of the penetration cell apparatus. However, the surface tension could likely change during liquid penetration through the test fabric while the fluid acclimates to alternate environmental conditions. These results provide the foundation to revise the ASTM F1670 standard to better characterize the measurement and handling of synthetic blood. This work also provides insight into factors to be considered when selecting an ASTM reference test fluid.

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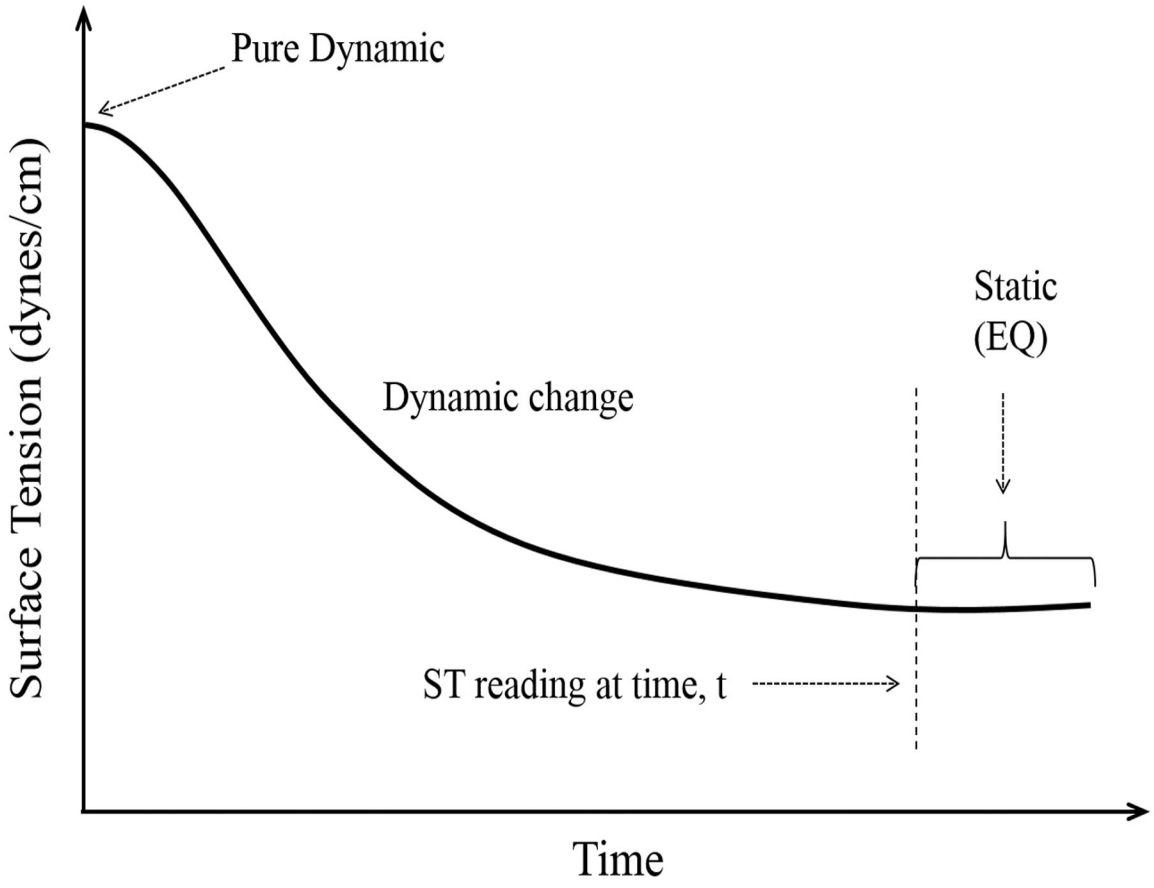
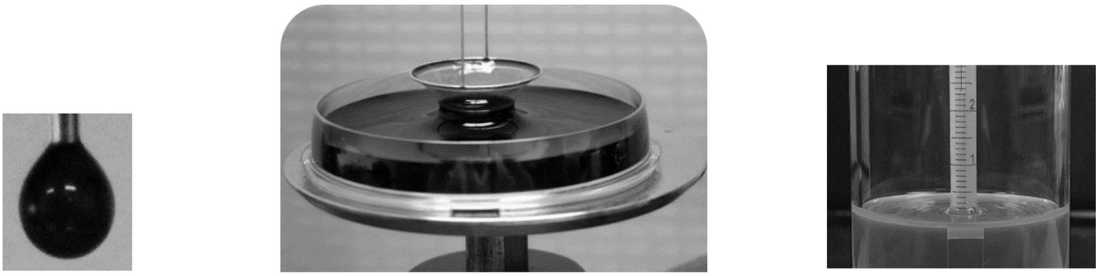


Figure 1.
Change in surface tension measured over time



a) Pendant Drop b) du Noüy Ring c) Capillary Rise

Figure 2.
Three methods used to measure surface tension

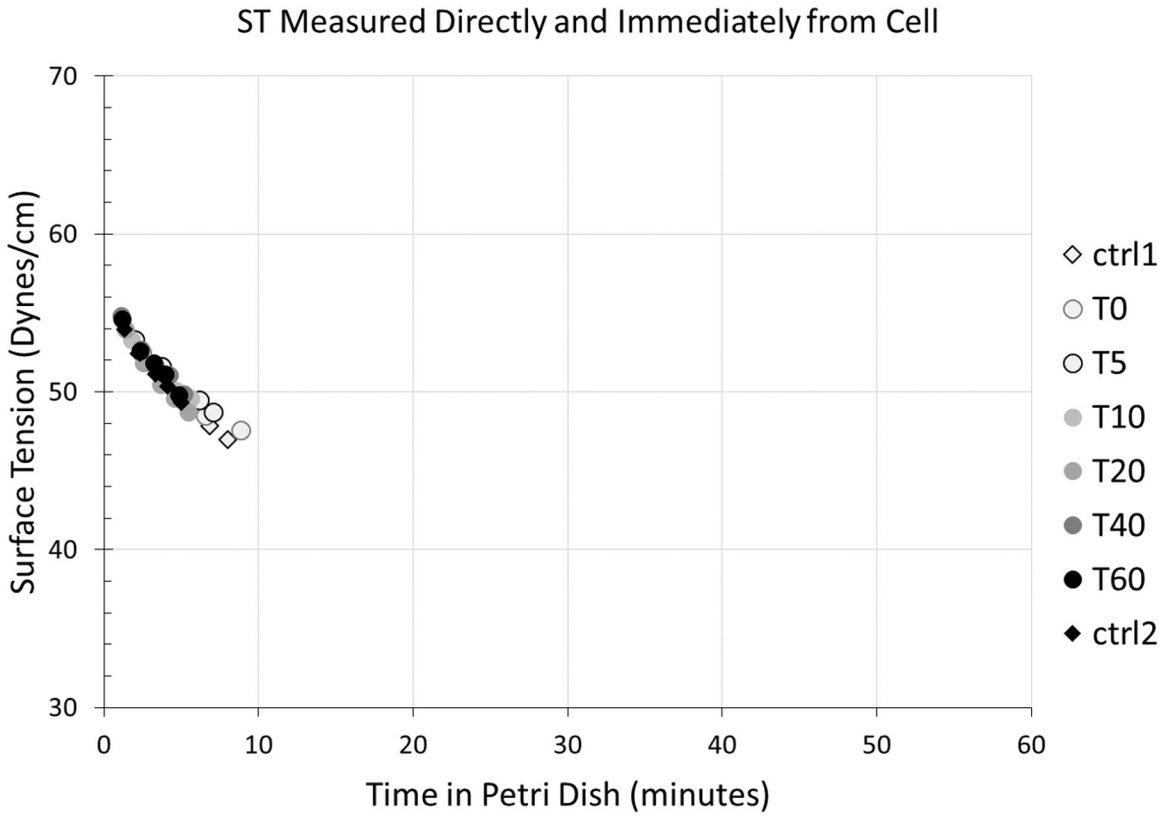


Figure 3.

Surface tension of synthetic blood at 22 ± 2°C measured with a dNR tensiometer with 7 mL of fluid in a 47-mm petri dish drawn from a penetration cell at six different times: 0, 5, 10, 20, 40, and 60 min (T0, T5, T10, T20, T40, and T60) and control measurements taken from bottle (Ctrl1 and Ctrl2). The penetration cell was in accordance with ASTM F903, Standard Test Method for Resistance of Materials Used in Protective Clothing to Penetration by Liquids

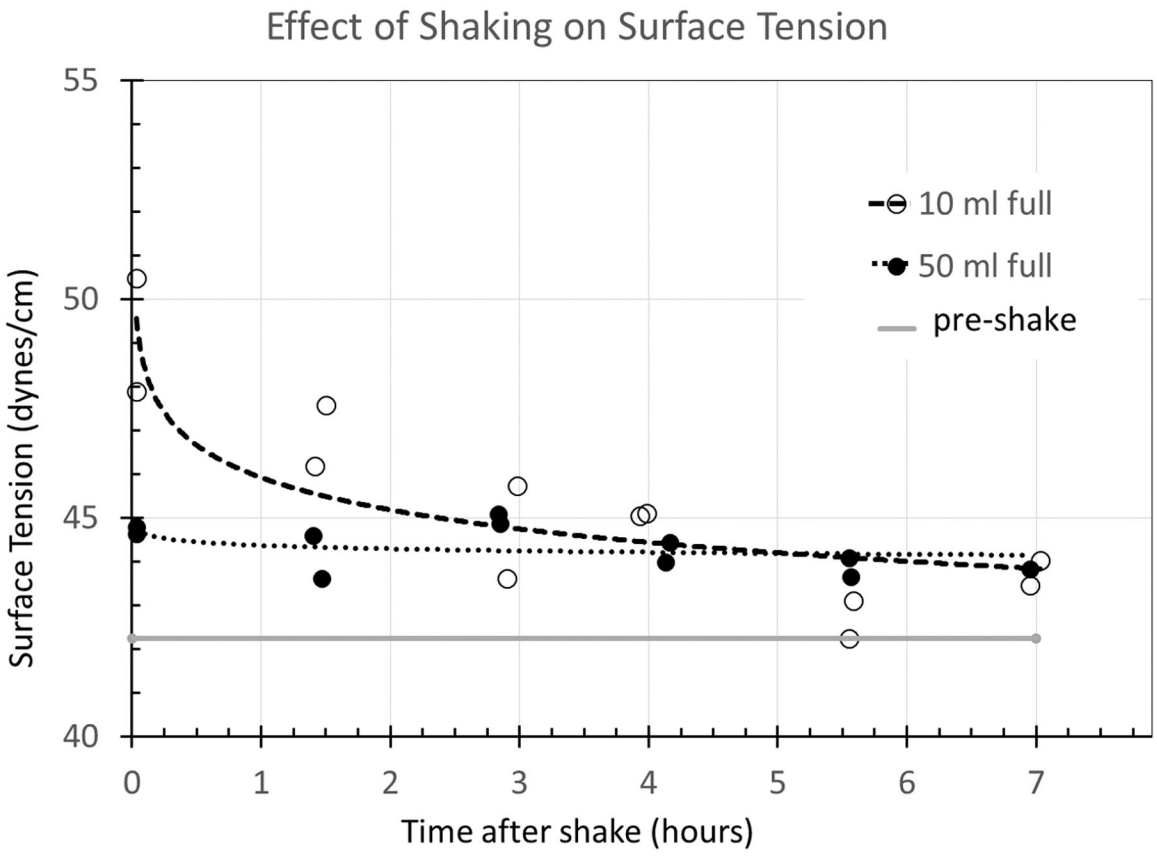


Figure 4.

Surface tension of synthetic blood at 22 ± 2°C measured with a du Noüy tensiometer with 20 mL of fluid in an 83-mm petri dish during 60 min in an undisturbed petri dish by two laboratories, NPPTL and Medline. Two laboratories by six times with three repeats in SE error bars

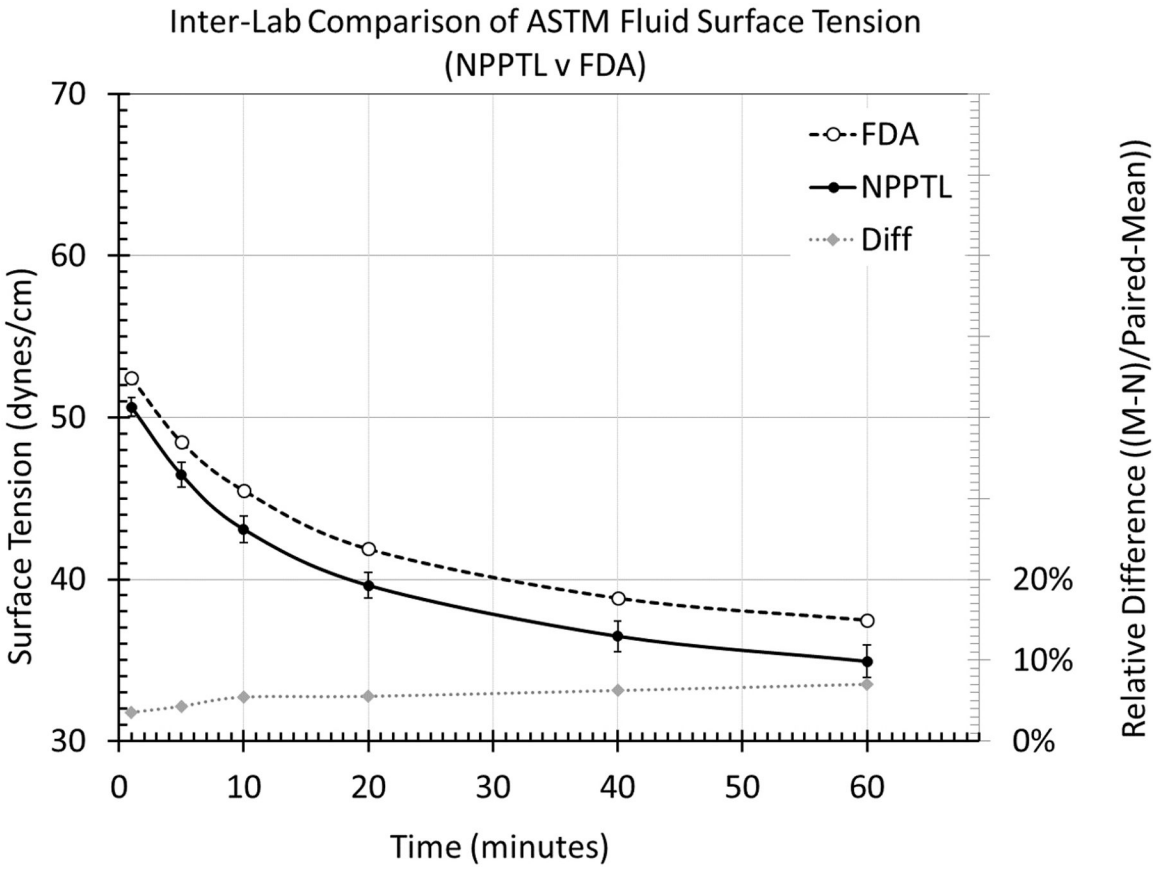


Figure 5.

Surface tension of synthetic blood at 22 ± 2°C measured with a du Noüy tensiometer with 35 mL of fluid in an 83-mm petri dish during 60 min in an undisturbed petri dish by two laboratories, NPPTL and FDA/CDRH. Two laboratories by six times with three repeats in SE error bars

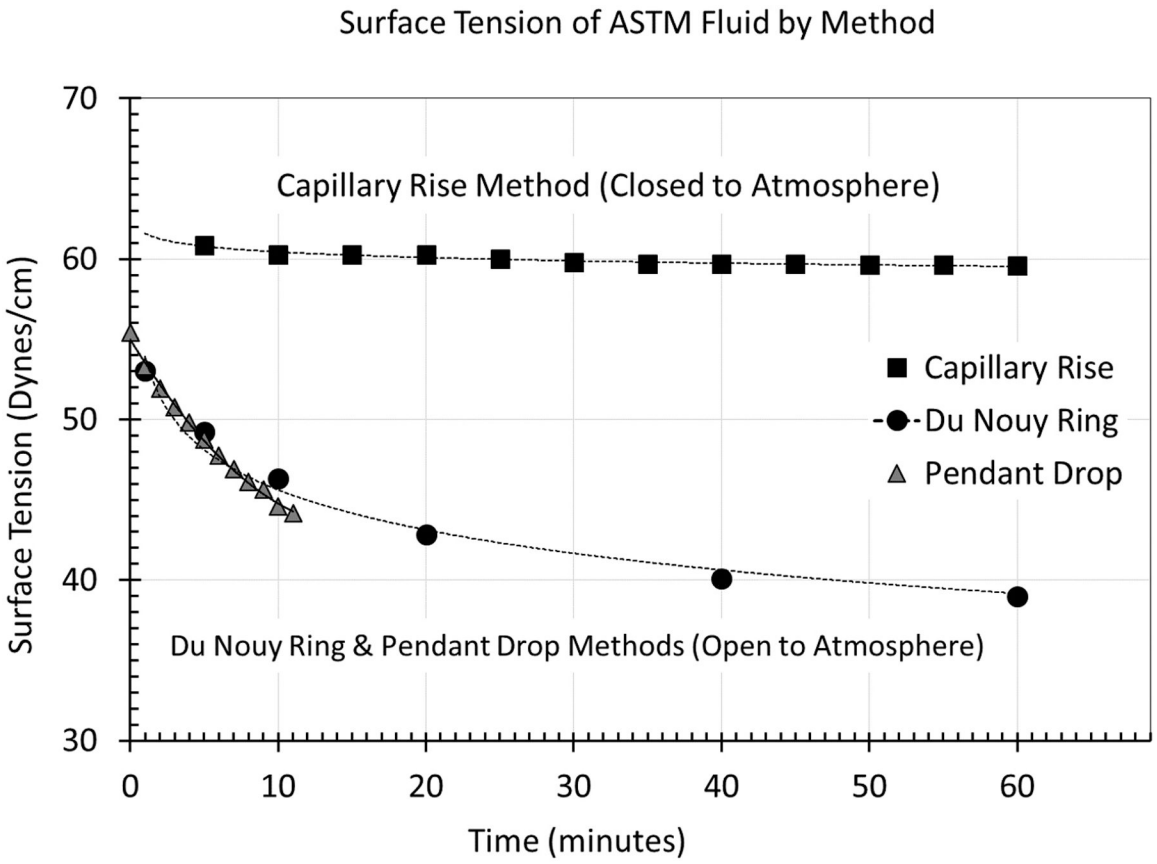


Figure 6.

Surface tension of synthetic blood at 22 ± 2°C measured by three different techniques: dNR method with 7 mL of fluid in a 47-mm petri dish during 60 min in an undisturbed petri dish, pendant drop method using Young–Laplace analysis mode, and a capillary rise method using a 0.5-mm capillary tube

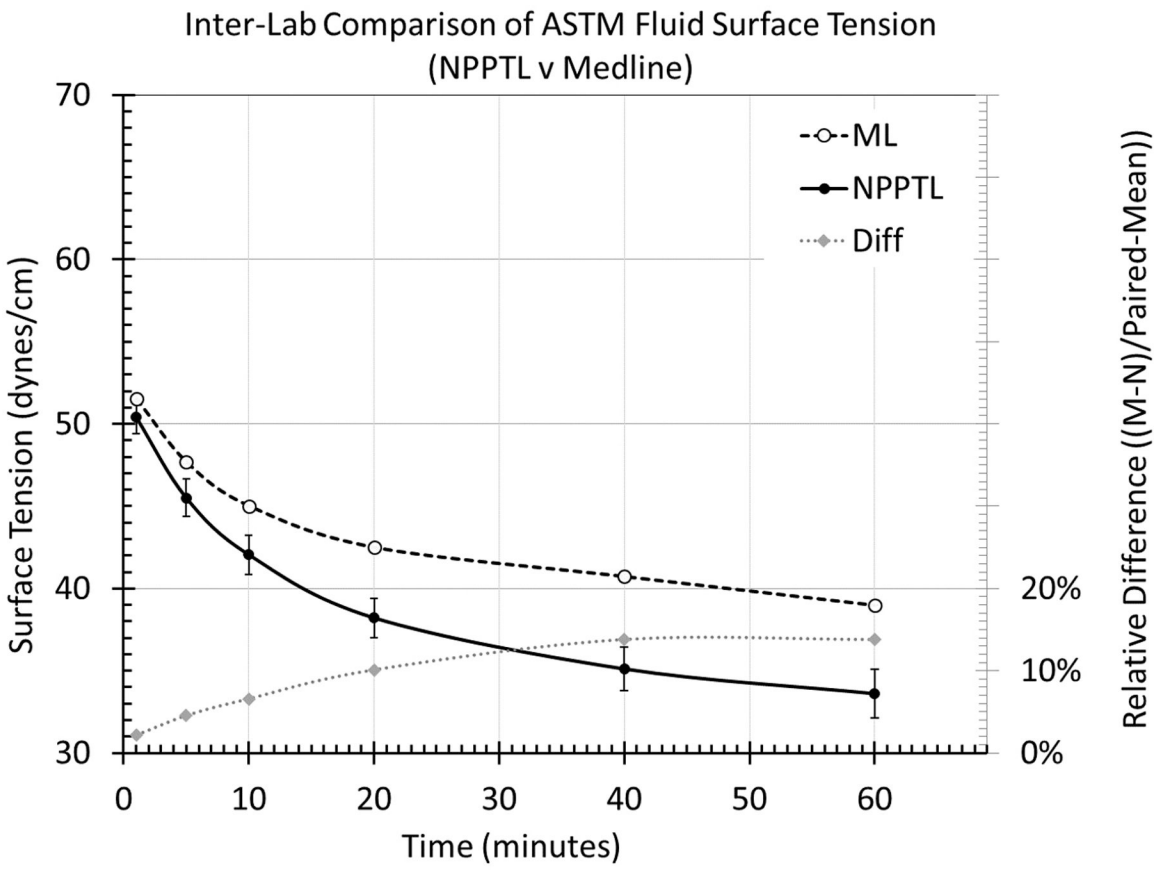


Figure 7.

Surface tension of synthetic blood at 22 ± 2°C measured with a du Noüy tensiometer with 7 mL of fluid in a 47-mm petri dish during 60 min in an undisturbed petri dish with two shaking treatments: 15% full and 95 % full. Two treatments by six times with three repeats with log fit curves

Table I.

Surface area to volume ratios (SA/V) of synthetic blood test configurations

Method	Container	Surface Area, cm^2	Volume, cm^3	SA/V, cm^{-1}
du Noüy ring	47 mm dish; 7 mL fluid	17.3	7	2.5
du Noüy ring	83 mm dish; 20 mL fluid	54.1	20	2.7
du Noüy ring	83 mm dish; 35 mL fluid	54.1	35	1.5
Pendant drop	15 μL drop	0.294	0.015	19.6
Capillary tube	0.5 mm capillary	0.0079	N/A	N/A