

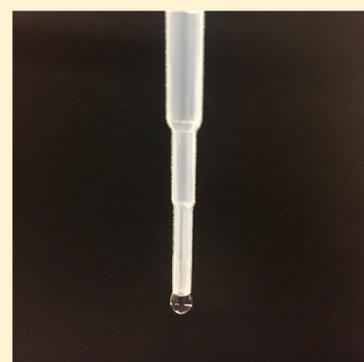
Use of Simplified Surface Tension Measurements To Determine Surface Excess: An Undergraduate Experiment

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S Supporting Information

ABSTRACT: The effect of surfactant adsorption on surface tension, as well as associated thermodynamic concepts are introduced in a laboratory experiment designed for undergraduate students. Using a reliable and accessible method, students measure the surface tension of aqueous solutions at different concentrations of sodium dodecyl sulfate. Students collect data to estimate the critical micelle concentration and quantitatively determine the maximum surface excess using the Gibbs adsorption equation. Students subsequently determine the surface area per molecule of this surfactant at the liquid–air interface and learn how to generate adsorption isotherm curves.



KEYWORDS: Hands-On Learning/Manipulatives, Laboratory Instruction, Chemical Engineering, Physical Properties, Micelles, Surface Science, First-Year Undergraduate/General, Second-Year Undergraduate

■ INTRODUCTION

The concepts of surface excess and the critical micellar concentration (CMC) are fundamental to the field of interfacial science and engineering. These concepts quantify the unique property of surfactants to adsorb at interfaces and to aggregate in surfactant solutions to form micelles. Experiments to determine the CMC and the surface excess as a function of bulk surfactant concentration are essential to student training. However, the measurement of these quantities often requires sensitive equipment and complex mathematical models. This can make it difficult to provide hands-on laboratory experiences for undergraduate students who are often taught in lab sections that have significant numbers of students. The availability of a sufficient number of duplicate experimental setups with sensitive equipment is often cost-prohibitive. Furthermore, the sensitive nature of such equipment often requires significant training time that may detract from the overall learning objectives that must be accomplished in the finite time allotted to a lab course. Thus, there is a need for laboratory experiments that are time-efficient, can be duplicated so all students can participate, and have results with sufficient accuracy that key concepts may be taught.

There are many undergraduate experiments that focus on the fundamental concepts of surface science in the literature.^{1–12} Dominguez et al.¹ determined the CMC of surfactant solutions using UV absorption spectroscopy, fluorescence spectroscopy, and electrical conductivity. Similarly, Huck-Iriart et al.² described an undergraduate experiment in which the surface tension of surfactant solutions is

obtained via a microscope equipped with digital image processing. Data are then used to illustrate concepts such as surface excess and adsorption isotherms. Castro et al.³ utilized the maximum bubble pressure (MBP) method to measure surface tension of surfactant solutions, and the data are plotted to determine the CMC. In that work, data are only used to determine the CMC, although the experiment could be extended to determine the surface excess of adsorbed surfactant. The MBP method is simple in concept, but the measurement device and software are quite expensive. Zhang et al.⁴ extracted surface tension versus concentration data and determined the surface excess and cross-sectional area of adsorbed surfactant. To do so, they utilized a specially constructed piece of equipment capable of measuring surface tension via multiple techniques, but the uniqueness of the equipment makes it difficult to duplicate. Bresler and Hagen⁵ documented an undergraduate experiment that determined the surface excess and the CMC with high precision,⁶ but the method utilized a DeNouy ring detachment method that requires a special tensiometer. Similarly, Marcolongo and Mirenda⁷ utilized electrical conductivity to measure these quantities, but the electrical measurements require special equipment and are limited to ionic surfactants. Meister and Latychevskaja⁸ utilized a pendant-drop method to measure surface tension, but did not use these data to determine the surface excess or the CMC. Alkawareek et al.⁹ used contact

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angle measurements to determine CMC; however, these measurements cannot be used to obtain surfactant adsorption properties (e.g., surface excess and surface area per molecule), and special equipment is required for these measurements, as well. Furthermore, there are other educational experiments that focus solely on surface tension measurement via specialized equipment, but surface excess or the CMC is not measured.^{10–12} All of the above-cited literature requires equipment that is not often available to large classes of undergraduate students, and some of the cited studies focus only on surface tension measurements alone with no further data analysis.

In this paper, we disclose an experiment that is appropriate for large classes of undergraduate students as it eliminates the need for expensive equipment and is easily duplicated. This method to measure surface tension and surfactant adsorption properties can be accomplished easily by students with high-school-level lab skills—yet the technique yields impressively accurate results. In addition, this experimental learning tool is designed so that the minimum number of data points is required to accomplish the intended objective, which is to obtain a reasonable estimate for the CMC, surface excess, and surface area per adsorbed molecule.

Here, students measure the surface tension of liquids with different surfactant concentrations by the “drop-weight” method, in which the mass of dispensed pendant droplets is measured and compared to the mass of similarly dispensed droplets of standard solutions with known surface tension. Once the surface tension data are collected, ancillary surfactant adsorption properties are extracted from the data. The overarching goals of the experiment are to impart to students an understanding of the impact of adsorbed surfactant on surface tension, to quantify this effect through the collection and interpretation of data, and to actively engage students in hands-on learning. The latter is important as it is well-understood that hands-on laboratory experiments enhance learning.¹³ Details on the assessment and attainment of key learning outcomes of the experiment are provided in this paper, as well.

■ BACKGROUND

Derivation of the Drop-Weight Equations Used To Extract Surface Tension

The drop-weight method has been established as a convenient method to determine the surface tension of liquids.¹⁴ The equation that relates the mass, m , of a pendant drop to the surface tension, γ , of the liquid and the radius, r , of the dispenser orifice is given by^{14,15}

$$mgf_c = 2\pi r\gamma \quad (1)$$

where g is the gravitational constant. In eq 1, f_c is a correction factor that accounts for the phenomenon that the full mass of a pendant drop does not detach from a dispenser. Specifically, f_c corrects for the mass measured during the experiments by increasing it to include the amount left behind on the dispenser. Consequently, the value of f_c must be larger than unity.

In deriving eq 1, it is assumed that the dispenser orifice is in full contact with the liquid so that the orifice diameter is equal to the cylindrical diameter at the top of the pendant drop at the point of release. If pendant drops from a standard solution

of known surface tension, γ_s , are carefully weighed, the correction factor can be found as

$$f_c = \frac{2\pi r\gamma_s}{m_s g} \quad (2)$$

where m_s is the mass of a dispensed pendant drop of the standard solution.^{15,16} This factor can then be used to find the surface tension of liquids with different surfactant concentrations from the mass of their drops and the radius of the dispenser orifice from eq 1. Alternatively, using the assumption of a constant correction factor, the surface tension can be calculated without the need to measure the orifice radius, r , if this radius is also constant. For this special case, the surface tension, γ_i , of the liquid of interest having drop mass, m_i , can be expressed in terms of the same quantities for a standard liquid by evaluating eq 1 for these quantities and dividing to obtain γ_i as

$$\gamma_i = \gamma_s \left(\frac{m_i}{m_s} \right) \quad (3)$$

In this paper, eq 3 is used to extract the dependence of surface tension on the concentration of the surfactant sodium dodecyl sulfate (SDS).

The validity of the assumption of constant f_c is demonstrated by the measured dependence of surface tension on SDS concentration, as well as the extracted surface excess; both do agree well with the literature values, as discussed in the [Results and Discussion](#) section. Although a surface tension and geometry-dependent correction factor is generally needed to extract properties suitable for academic studies, it is not needed here within the desired accuracy of the experiments—which makes the experiment accessible to the target undergraduate audience.¹⁷

■ EXPERIMENTAL SECTION

Methods

The experiment reported here and experimental results to follow were performed by 48 students in a second year undergraduate chemical engineering laboratory course entitled Chemical Engineering Principles Lab (CHME-391). This two-credit course (one semester) comprised 10 different modules covering important topics in chemical engineering. The duration of each module varies depending on the learning objectives. The Surface Science module consisted of four class periods lasting approximately 3 h each with the following activities. The class of 48 students was divided into 17 groups of 2–3 students each and was taught in two sections. In each section, a detailed syllabus was provided to the students and an overview of the module was described (see “Surface science module description & syllabus” in the [Supporting Information](#) (SI)). Prior to the lab portion of the module, six introductory lectures on surface tension, adsorption isotherms, surfactants, and other related topics were given to provide context (see “Surface science module lectures” in the SI for these six lectures). The lectures included lab demonstrations, video demonstrations, and “fun” experiments and were presented during the first three classes. The experiment described herein was carried out during the last class (3 h length). A detailed lab procedure was given to the students (see instructions in the SI), was reviewed with them for questions, and then the experiment proceeded as follows.

Each group was provided with a table of surface tension values for pure water as a function of temperature. The temperature in the room was noted, and the corresponding reference surface tension value was recorded. For the day of the experiment, the students used a standard surface tension value of 72 mN/m for a recorded room temperature of 25 °C. The average drop mass for solutions of SDS was determined as described below. Pure distilled water (18.3 mΩ) was used as the standard liquid of reference. Students prepared stock solutions of 0.05 and 0.01 M from a concentrated SDS solution provided (0.1 M). Then they diluted them to make a series of concentrations between 0.0001 and 0.05 M. Note that the dilutions were made by volume, and not mass, as the concentrations of the solutions were low.

The mass of several empty vials with their caps was measured and recorded, and the vials were individually labeled to denote the solution/reference standard that would be collected. A pipet was used to dispense multiple drops of each solution into the corresponding vials, and the number of drops per vial was recorded (for details regarding the pipet used see discussion below). Vials were capped immediately after drop-dispensing to minimize the effects of evaporation. The mass of each vial was then remeasured, and the mass of the liquid was determined by subtracting off the masses for each empty vial. The average drop mass was calculated by dividing the liquid mass in each vial by the corresponding number of drops used. The average drop masses (obtained for surfactant and standard solutions) were substituted into eq 3 to obtain surface tension values for each solution. Note that the procedure to preweigh and label the vials was adopted in order to accommodate multiple groups of students with the two available analytical balances in the lab (Mettler Toledo Newclassic MS balances with a precision to 0.1 mg).

To minimize the contribution to variability attributed to drop detachment, a standardized drop-formation procedure was adopted. The samples were measured by one individual per experimental group. The same disposable pipet was used for all the experimental measurements to eliminate variability in the pipet orifice radius. Plastic pipets (Fisherbrand, disposable, polyethylene transfer pipets, cat. #13-711-9AM from Fisher Scientific) were used to prevent wetting of the outside edge of the pipet. Preliminary experiments with thin glass pipets revealed significant wetting on their outer surface, which led to increased variability in drop masses. Visual observation (no magnification) of the drop detachment from the polyethylene transfer pipets confirmed this nonwetting behavior, therefore, the radius to be used in eqs 2 and 3 was that of the dispenser orifice. These pipets had the added benefit of being safer to use. Additionally, it was observed that the drops detached when their tangents were vertical and parallel to the centerline axis of the pipet (the pipet was held vertically as discussed below). Because the same pipet was used for all samples, the measurements progressed in the order of increasing concentration—starting from the reference sample—to minimize contamination error.

Students were instructed to form pendant drops slowly to provide enough time for the surfactant to fully adsorb to the air–liquid interface before dispensing. If dispensed too quickly, the extracted measurements would not be the true static surface tension, as the interface would not achieve an equilibrium with its bulk concentration. Once formed, students were told to hold the pendant drop for a few seconds in its critical configuration prior to detachment; previous studies^{18,19}

suggest that a few seconds is sufficient to achieve equilibrium. With the manual dispensing method used, students did find it difficult to maintain pendant drops at the final critical configuration for longer times. As discussed further in the [Results and Discussion](#) section, this limitation may have caused some minor errors in the final results. Nevertheless, the results obtained demonstrate that the magnitude of such errors was not sufficient to invalidate the simplified experiment within the scope of our educational objectives.

It was also suggested that students begin each sample with “practice drops” and discard the first droplets that were formed from the pipet. Such initial droplets were often observed to include air bubbles that would introduce error in the drop mass.

Another experimental concern was to minimize variations in the orientation of the axis of the pipet—which could invalidate the assumption of constant correction factor underlying eq 3. Drops needed to be consistently dispensed with the axis of the pipet perpendicular to the bottom of the vial. According to experiments done by Gans and Harkins, the effect on drop mass from an axis angle deviation under 2° is negligible.²⁰ They argue that because a tilt of such magnitude is noticeable to the human eye, the drop masses used to measure surface tension are accurate if no tilt is perceived without magnification. The students were indeed instructed to keep the pipet axis vertical and, if necessary, to find a reference edge on the lab bench (such as the wall of a beaker) to look at while aligning the pipet before dispensing a drop.

Materials

The SDS was purchased from Sigma-Aldrich (cat. #436143-100G with ACS reagent grade purity of 99% or higher). It is widely accepted that when SDS is used, purification such as by recrystallization may increase the surface tension values obtained.²¹ In such cases, the impurity responsible for the surface tension decrease is believed to be dodecanol. In the presence of this impurity, a minimum in the surface tension as a function of concentration is observed around the CMC. When dodecanol contamination is present, it is believed that it decreases the surface tension at concentrations below the CMC. However, for concentrations higher than CMC, the dodecanol is solubilized by the micelles, thus eliminating its effect, increasing the surface tension, and creating the minimum. No such minimum was observed with the SDS used in these experiments, so no purification of the purchased SDS was deemed necessary. In addition, all the SDS solutions used were fresh to avoid the hydrolysis of any SDS to dodecanol.

Hazards

Although sodium dodecyl sulfate is relatively safe at low concentrations, it is a flammable solid. The powdered solid is orally toxic and can cause serious eye damage. Gloves and safety glasses should be worn at all times when handling any SDS solution, as it is an eye and skin irritant.²²

RESULTS AND DISCUSSION

Student groups determined the surface tensions of seven solutions containing SDS using eq 3. Typical data from one student group are provided in [Table 1](#); students subsequently plotted these data, as illustrated in [Figure 1](#). The students examined this plot and applied the learnings from the lecture portion of this lab module (see lecture material in the [SI](#)) to determine the CMC for this surfactant and then estimate the

Table 1. Typical Student Results for Surface Tension

concentration (M)	average mass of 20 drops (g)	surface tension ^a (mN/m)
1.0×10^{-4}	0.833	73.68
5.0×10^{-4}	0.785	69.43
1.0×10^{-3}	0.775	68.55
2.5×10^{-3}	0.739	62.60
5.0×10^{-3}	0.592	52.36
1.0×10^{-2}	0.485	42.90
5.0×10^{-2}	0.491	43.43

^aCalculated surface tension using eq 3.

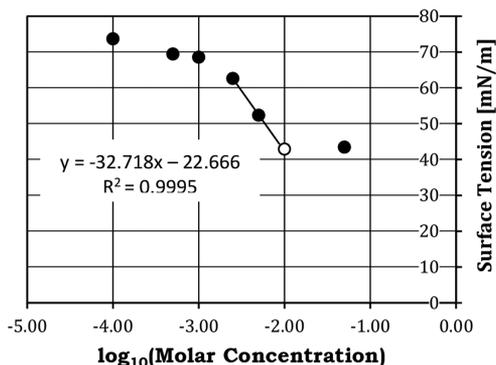


Figure 1. Typical student-generated surface tension plot. The open plot symbol in the figure provides an estimate of the critical micellar concentration at 10^{-2} M. The solid line is the best fit to the three data points it intersects. The slope of this line has a value of -32.718 mN/m, which is used to determine $d\gamma/d \log_{10}(c)$ in eq 4.

maximum surface excess. Based on these learnings, the CMC was determined by students as the lowest concentration at which the lowest surface tension was measured on a surface tension plot, as indicated by the open plot symbol in Figure 1. Furthermore, a quantitative value of the maximum surface excess was extracted from Figure 1 by noting that it occurs in the linearly sloped region of the plot just below the CMC. In accordance with Gibb's adsorption equation for an ionic surfactant, the surface excess, Γ_v , is given by

$$\Gamma_i = -\frac{1}{4.60SRT} \frac{d\gamma}{d[\log_{10}(c)]} \quad (4)$$

where R is the ideal gas constant in units of $\text{erg K}^{-1} \text{mol}^{-1}$, T is the absolute temperature in Kelvin, γ is the surface tension in mN/m, and c is the concentration in mol/L.⁵ Each student group used the linear portion of the data in Figure 1 to determine the best-fit slope $d\gamma/d[\log_{10}(c)]$ of that curve.

Once the surface excess was determined from eq 4, each group also extracted the area per molecule through the relationship:

$$A^\Gamma = \frac{1}{\Gamma_v N_{\text{Av}}} \quad (5)$$

where N_{Av} is Avogadro's number. Figure 2 compiles all the surface tension versus concentration data collected from the groups in this experiment. As evidenced in Figure 2, the average surface tension data collected by the students follow the literature values for the surfactant with reasonable accuracy.²³

The extracted results for surface excess and surface area per molecule (eq 4 and eq 5) obtained by the students are

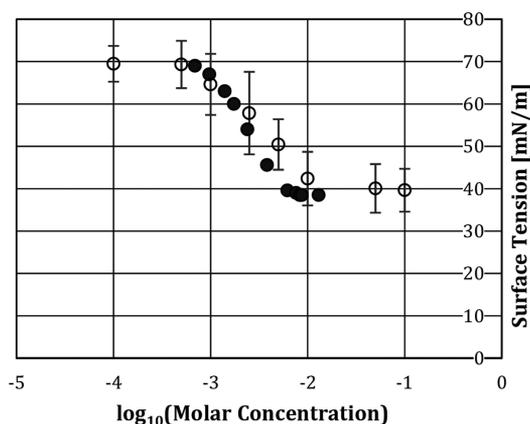


Figure 2. Average surface tension for both lab sections (open circles) as compared to reference literature values (solid circles).²³ Error bars represent a confidence interval of 1 standard deviation of the experimental values.

summarized in Table 2 and compared with accepted values provided by Rosen.²⁴

Table 2. Comparison of Values of Surface Excess and Molecular Surface Area

source	surface excess ^b (mol/cm ²)	molecular surface area ^b (Å ² /molecule)
section 1	$2.74 \times 10^{-10} \pm 0.75 \times 10^{-10}$	62.7 ± 20.1
section 2	$2.67 \times 10^{-10} \pm 0.80 \times 10^{-10}$	67.8 ± 22.4
literature ^a	3.1×10^{-10}	53

^aSee ref 24. ^bValues were determined by averaging the values obtained from each group. The variability in the measurements is expressed as 1 standard deviation from the average value.

The underlying student data contributing to the averages in Table 2 are provided in Figure 3. The data indicate a systematic error by both lab sections as evidenced by the nonrandom distribution of the data around accepted values. The origin of this error is apparent by inspection of Figure 2, where the slope of the student data just below the CMC is not as steep as the corresponding slope exhibited by the literature data.²³ This deviation produces a decrease in the surface excess value (Figure 3a) and an equivalent increase in the surface area per molecule value (Figure 3b). A possible explanation for this result is that surfactant is not fully adsorbed to the air–drop interface. To dispense a drop, students apply pressure to the bulb of a pipet via their fingers. It is difficult to maintain a drop in its critical configuration before detachment for a significant length of time using manual pressure. Thus, drops likely detach before an equilibrium surface adsorption is achieved, and this could explain the observed deviation.²⁵

Additionally, a general discussion regarding the origin of the surface tension versus concentration curve—including the micellar region—was provided to students during the lab as auxiliary instructional material. The students were also shown how to use eq 4, along with the surface tension versus \log_{10} of concentration curve shown in Figure 1, to generate an adsorption isotherm in the form of surface excess versus concentration (see slide 61 of Surface Science Module Lectures in the SI). This could be done by extracting the local slope of the curve in Figure 1 at various concentrations. However, as the number of concentrations studied was small in

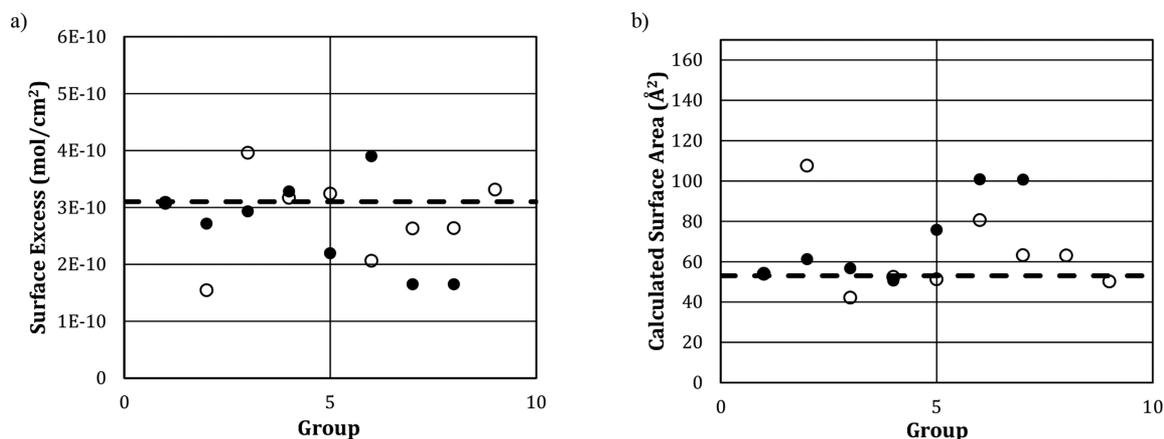


Figure 3. Surface excess and surface area per molecule reported for each student group. (a) Surface excess (mol/cm²) compared with its literature value at 25 °C, represented by the dashed line.²⁴ The open and solid circles in the plot are data for sections 1 and 2, respectively. (b) Surface area per molecule (Å²) compared with its literature value at 25 °C, represented by the dashed line.²⁴ The open and solid circles in the plot are data for sections 1 and 2, respectively.

order to make the experiment fit within time allotted for the lab, there was not enough resolution in the Figure 1 curve to obtain reasonably accurate slopes except in the linear region of the figure. Thus, only the maximum surface excess, which corresponds to that linear region, was extracted in the experiment.

At the end of the experiment, each group of three students was required to submit a Microsoft Excel file with all their data, calculations, and observational comments. These results were summarized in the form of a short technical report submitted for the team. Each student was also graded individually on key concepts taught in the module via three quizzes. The final grade for the Surface Science module of the laboratory course was obtained as a weighted average of these component grades.

The success of this experiment and the supporting lectures in achieving the learning objectives of the Surface Science module was assessed by three criteria: (1) the accuracy of the reported values of the CMC, the maximum surface excess just below the CMC, and the area per molecule of the adsorbed surfactant at this bulk surfactant concentration (see Microsoft Excel file containing typical experimental data in the SI); (2) the understanding of the concepts of surfactant adsorption, micelle formation, surface excess, and adsorption isotherms as reflected by the submitted technical reports; and (3) the level of understanding of these same concepts as reflected by the answers to three quizzes related to these concepts (see these quizzes in the SI).

Student performance indicated that the learning objectives were achieved based on the final average grade of 88% for the lab module (see grade book for the module in SI). Thus, it is concluded that the experiment described herein is a good instructional tool for teaching fundamental surface science concepts to second year students in the Chemical Engineering program. In addition, student evaluations for the Surface Science portion of this course were quite positive, and this indicated that the students were receptive to the experiment and analysis of their data. It is worth noting that a recent article²⁶ has confirmed that the Gibbs adsorption method used in this experiment (see eq 4) does estimate accurately the surface excess for surfactant concentrations that are lower than the CMC. This demonstration further confirms the soundness of this educational experiment.

CONCLUSIONS

The experiment described in this paper provides a simple means to introduce the thermodynamic concepts of surfactant adsorption to undergraduate students. Results were generated for an SDS–water solution by undergraduate students as part of a Surface Science module in a second year laboratory course. In spite of its simplicity, the experiment yielded surface tension vs SDS concentration curves, as well as extracted surface excess and area per surfactant molecule, close to those reported in the literature. The experiment itself was imbedded in an overall lab module that included lectures, quizzes, and an experimental lab report. Learning objectives were met based on student performance on these evaluation components. It was thus concluded that this experiment, and the module as a whole, is an effective introduction to key elements of surface science.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.8b00667.

Instructions for the SDS adsorption experiment (PDF, DOC)

Quizzes 1–3 (PDF, DOCX)

Answer key for quizzes 1–3 (PDF, DOCX)

Surface science module description and syllabus (PDF, DOCX)

Surface science module lectures (PDF)

Experimental data example (XLSX)

Grade book for the module (XLSX)

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Notes

The authors declare no competing financial interest.

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