

Water on a Hydrophobic Surface: A Comparison of Contact Angle Versus Depletion Layer Using Surface Plasmon Resonance

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Second Reader: Dr. Shafiqur Rahman

I hereby recognize and pledge to fulfill my responsibilities, as defined by the Honor Code, and to maintain the integrity of both myself and the Allegheny College community as a whole.

Pledge _____

Jessica L. Jeroski

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1. Abstract

This research looks at how water interacts with a hydrophobic surface. The degree of hydrophobicity of a surface is determined by measuring the contact angle between the water and the surface. An interruption in the network of hydrogen bonds causes water to be repelled when in contact with a surface that is hydrophobic. Conversely, a hydrophilic surface has a small contact angle and a does not repel water. It is expected that a region of depleted density, called a depletion layer will form near a hydrophobic surface, and that the depletion layer will be larger for surfaces with higher degrees of hydrophobicity.

The techniques of surface plasmon resonance are used to investigate the interaction between water and a surface. Gold-coated glass slides can be adjusted to various degrees of hydrophobicity using octadecanethiol (ODT), a hydrophobic organothiol, or 11-mercapto-undecanoic acid (MUA), a hydrophilic organothiol, or some mixture of the two. The different mixed solutions create a range of contact angles that are used to find the relationship between contact angle and depletion layer. By comparing experimental and theoretical data, the thickness of the depletion layer is determined,

The data for this experiment proved inconclusive, potentially due to the inherent roughness of the coating mixtures and the instability of the self-assembled monolayers of organothiols. Further research will be needed to determine an exact relationship between contact angle and the resultant depletion layer.

2. Introduction

2.1 Background

Water is one of the most plentiful substances on the Earth. It covers approximately two-thirds of the surface of the planet and makes up almost 70% of the human body by volume [1]. People are surrounded by water on a daily basis and yet there is still much to be learned about it. Water is an extremely complicated fluid that is held together by the formation of a three-dimensional network of hydrogen bonds, the strongest type of intermolecular chemical bonds (see Figure 1).

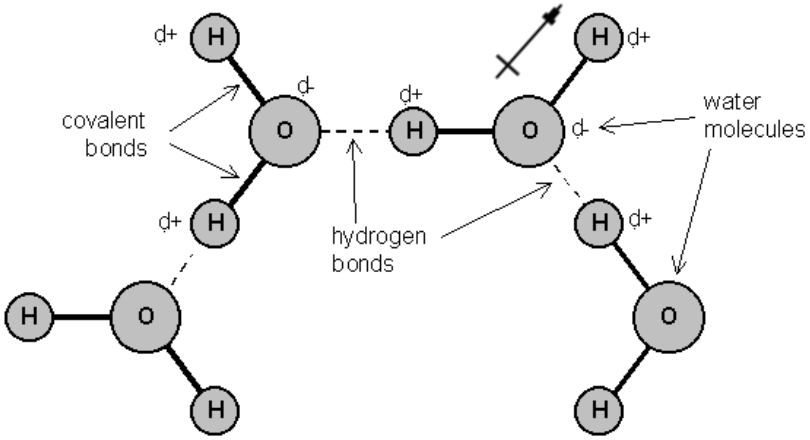


Figure 1. Molecular Structure of Four Water Molecules
Water is held together with hydrogen bonds between the oxygen atom of one molecule and the hydrogen atom of another. The slightly positive hydrogen ends, shown by the d+, orient themselves closest to the slightly negative oxygen ends, shown by the d-.
<http://www2.lbsdk12.com/science/Anatomy/anatomyunit201.html>

Hydrophobicity is the interruption of the network of hydrogen bonds, caused by a surface in contact with water and so the surface appears to repel the water. Hydrophobicity is a combination of two Greek words: “hydro”, meaning water, and “phobos,” meaning fear [2]. At a hydrophobic surface, it is energetically favorable for water to bond with itself via hydrogen

bonds rather than the surface via a weaker type of bonding, and so the water tries to minimize contact with the surface and thus creates beads or droplets.

2.1 Applications

The study of hydrophobic surfaces is applicable to many interactions in our day-to-day life. Examples of hydrophobic surfaces include the morning dew on plants, stain-resistant clothing, and non-stick cookware. Scientific applications of this study include protein folding within the human body, colloidal-self assembly, uses in fluid engineering, and drug delivery [2][3].

2.3 Degree of Hydrophobicity (Contact Angle)

The degree of hydrophobicity of a surface is defined by the contact angle created by the surface and the tangent to the drop of water at the surface, as seen in Figure 2. When the angle produced is greater than 90 degrees, the surface is considered hydrophobic, and when the angle is less than 90 degrees, it is considered hydrophilic, meaning "water-loving" [2].

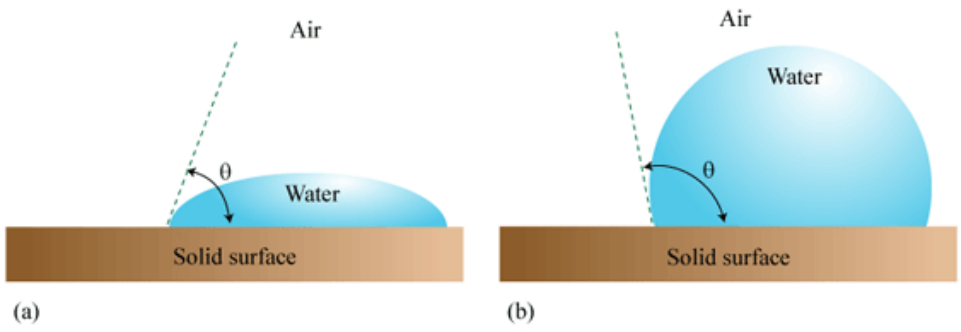


Figure 2. Water Drops with Different Contact Angles on a Surface
In the picture, (a) shows a hydrophilic surface with a contact angle, θ, less than 90 degrees, and (b) shows a hydrophobic surface with a contact angle greater than 90 degrees.
<http://www.hk-phy.org/atomic_world/lotus/lotus02_e.html>

This experiment uses hydrophobic and hydrophilic surfaces on gold-coated slides with a range of different contact angles. The surfaces are created using self-assembled monolayers (SAMs) of different organothiols. Octadecanethiol-1 (ODT), which has a contact angle of approximately 100 degrees, is used to form hydrophobic surfaces. Hydrophilic surfaces are made using SAMs of 11-mercapto undecanioc acid (MUA), which has a contact angle of approximately 30 degrees. A range of angles was achieved by creating mixtures of these different solutions in varying proportions. The angles used for the tests are shown in Table 1, below, along with the determined mixture for each contact angle.

Averaged Contact Angle, θ	% MUA	% ODT
30°	100	-
102.2°	-	100
79.9°	35	65
61.9°	80	20

Table 1. Table of Different Solutions and Their Respective Contact Angles
The table lists the different contact angles for various concentrations of hydrophobic and hydrophilic solutions used in this experiment. Error for the measurement of contact angles is approximately two degrees.

2.4 Depletion Layer

When an extended hydrophobic surface comes into contact with a large quantity of water, there is an interruption in the network of hydrogen bonds. However, the presence of the bulk water does not allow for the formation of individual droplets of water. The surface tries to repel the water in order to minimize contact, and as a result a region of low density, called a depletion layer, is formed at the surface only a few angstroms thick (see Figure 3) [2][4][5].

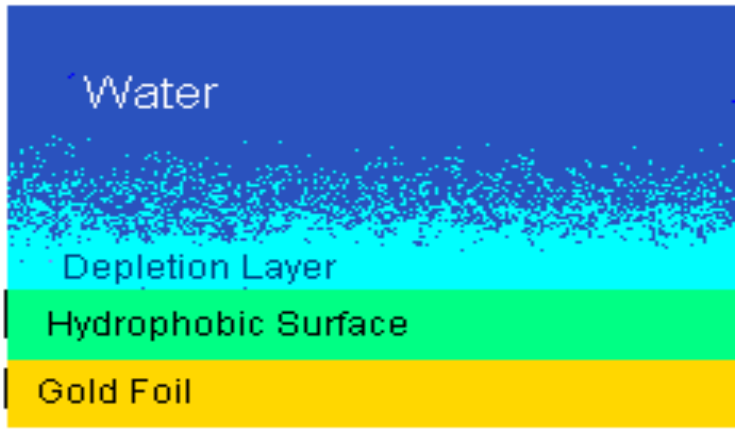


Figure 3. Surface Layers of Study

The gold-coated slide is covered with a hydrophobic (or hydrophilic) solution and when water comes into contact with it, a low-density depletion layer forms.

Image by Poynor.

3. Previous Research

Hydrophobicity and its effects have been studied for many years. In 1973, Stillinger presented the idea of a region of lower density between water and a hydrophobic surface. Using scaled-particle theory, he proposed that a thin layer of water vapor would surround large hydrophobic objects due to an interruption in the hydrogen-bonding network where the water met the surface. This vapor layer would have a density 50,000 times less than that of bulk water. For water around small particles of a hydrophobic nature, the water molecules would reorganize themselves in order to maintain their bonds around the hydrophobic particles [4].

More recently, in 1999, Lum, Chandler, and Weeks used Stillinger's in order to test if there is a difference between the interaction of water and a hydrophobic surface at different scale lengths. The results for small hydrophobic particles showed that, on the nanometer scale, water droplets arrange in such a way as to keep their hydrogen-bonding network intact and maintain a consistent density, in agreement with Stillinger. However, at a larger length scale, it is impossible for water to maintain a consistent density. The crossover between these two phenomena happens at the length scale of nanometers. Their simulation was completed such that it could be applicable to not just water, but any confined fluid [5].

In 2006, Poynor et al. tested the theory that nanobubbles, or tiny nanometer-scale sized pockets of air, exist on the surface of a hydrophobic material. Using x-ray reflectivity, they were able to disprove the presence of nanobubbles and to show that there instead exists a depletion layer of lower density. The layer exists at a thickness of two to four angstroms when using octadecylsilane as a hydrophobic surface, with contact angles ranging from 97 to 110 degrees [6].

In 2010, Chattopadhyay et al. used the results of the 2006 experiment by Poynor et al. to study the relationship between the degree of hydrophobicity of a surface, as quantized by the contact angle, and the resultant depletion layer, attempting to find a relationship between the two quantities [6][7]. Combining the results for angles between 105 and 110 degrees from experiments by others with their own results, Chattopadhyay et al. were able to show a linear relationship, within error, between the contact angle and the depletion layer, as seen in Figure 4 [5][7][8][9].

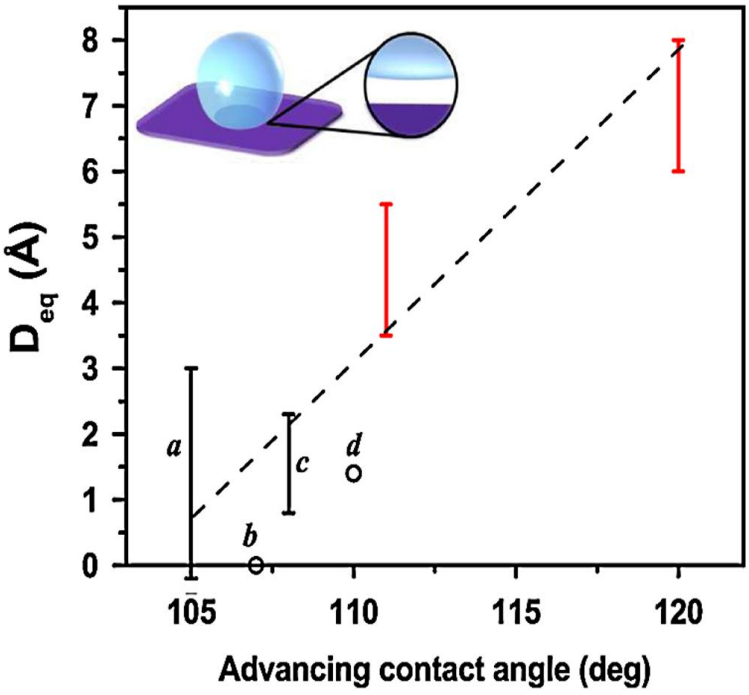


Figure 4. Results from experiment by Chattopadhyay et al.
This graph shows the relationship between depletion layer, in angstroms, and contact angle, in degrees. (a): for partially deuterated alkanethiol SAMs; (b): for octadecyltrichlorosilane SAMs (error bars not reported); (c): from Ref. [6], for octadecyltriethoxysilane SAMs; (d): for FAS17 SAMs (error bars not reported). The two unlettered points are results of the experiment by Chattopadhyay et al. for FAS13 and FAS25, two fabricated fluoroalkylsilane SAMs. Experiments (a), (b), and (d) referenced in [7].
Image by Chattopadhyay et al.

In the fall of 2011, Caitlyn Neidig began research in Poynor's lab to further study the relationship between contact angle and depletion layer. Though she attempted to test a range of angles between 40 and 105 degrees in order to further the data included in Figure 4, the various solutions did not give the expected contact angles, and only angles less than 50 degrees and greater than 95 degrees were actually studied. Results from Neidig's work are seen in Figure 5.

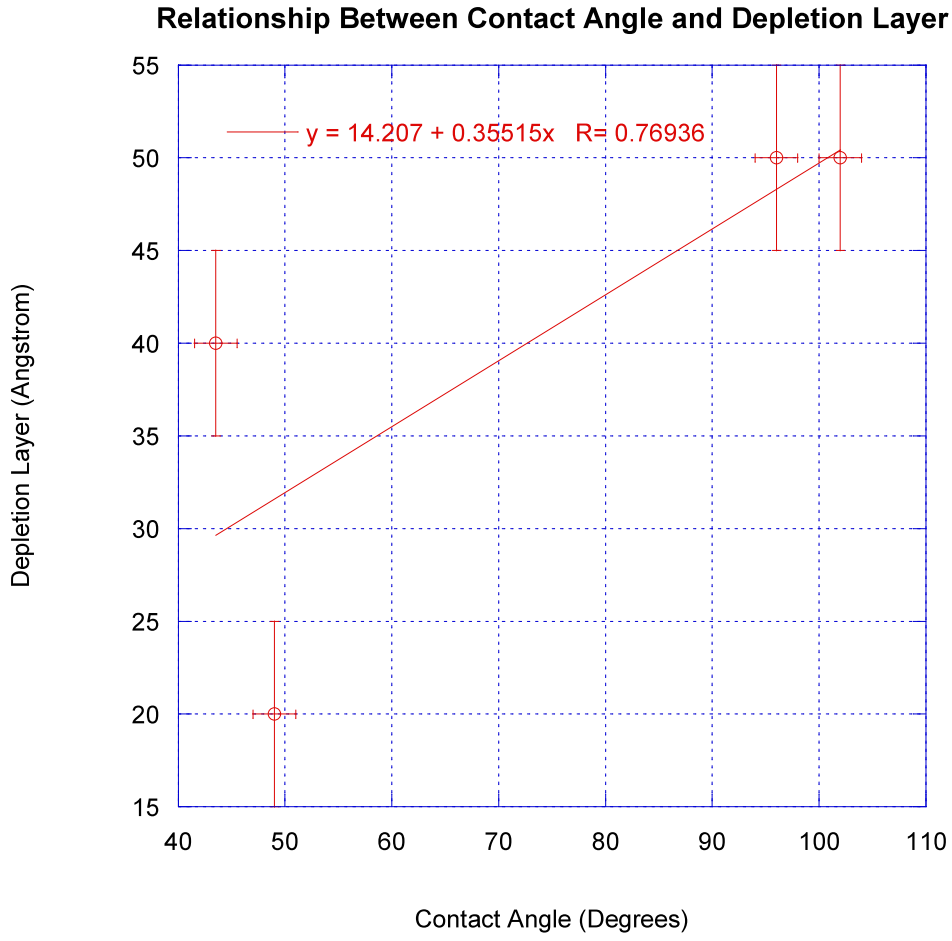


Figure 5. Results from Experiment by Neidig

The above graph shows the results of the experiment by Neidig. She continued the work done by Chattopadhyay et al. in an attempt to find a definitive relationship between the contact angle and the depletion layer. The vertical error bars show the error within the depletion layer thickness and the horizontal error bars show the standard deviation error from measuring the contact angles of the slides.

The focus of this work was to determine a definite relationship between the contact angle and the thickness of the resultant depletion layer when water is in contact with a hydrophobic surface. By mixing different concentrations of ODT and MUA, an indicative range of angles between 30 and 100 degrees can be studied.

4. Theory

4.1 Total Internal Reflection

In studying the effects of hydrophobic surfaces with a coherent form of light such as a laser, it is necessary to understand certain properties of optics. When light strikes a surface, the ray is either reflected, refracted, or both. The amount of light refracted is based upon the Fresnel Equations for p-polarized and s-polarized light, given by

$$R_p = \left(\frac{n_2 \cos\theta_1 - n_1 \cos\theta_2}{n_2 \cos\theta_1 + n_1 \cos\theta_2} \right)^2 \quad (1)$$

$$\text{and} \quad R_S = \left(\frac{n_1 \cos\theta_1 - n_2 \cos\theta_2}{n_1 \cos\theta_1 + n_2 \cos\theta_2} \right)^2 \quad (2),$$

respectively, where n_1 and n_2 are the indices of refraction for the surfaces, θ_1 is the angle of incidence, and θ_2 is the angle of transmittance [8]. Figure 6 shows the polarizations of laser light.

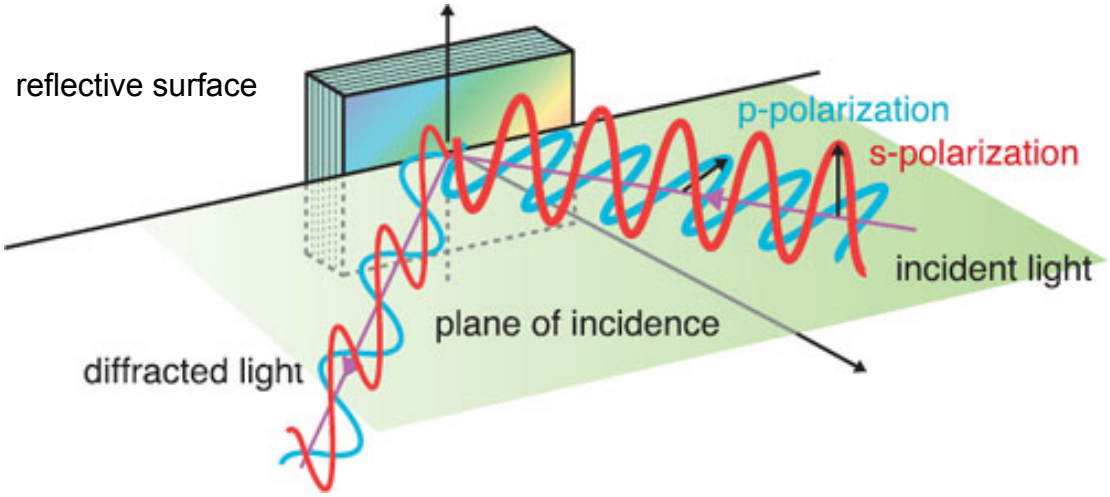


Figure 6. Diagram of S- and P-polarized Light Incident Upon a Reflective Surface
As shown, p-polarized light is parallel to the plane of incidence and s-polarized light is perpendicular to the plane of incidence.
<http://jolisfukyu.tokai-sc.jaea.go.jp/fukyu/tayu/ACT05E/05/0504.htm>

In this experiment, the laser emits p-polarized light, in which the waves of oscillation are parallel to the plane made by the direction of propagation, so the reflectance from Equation 1 is used.

If the angle created by the incoming ray of light and the surface, called the incident angle, is less than the critical angle, then light is both reflected and refracted. When the incident angle is greater than this angle, all of the light is reflected, leading to a phenomenon called total internal reflection [9][10]. The angle at which this occurs can be determined using Snell's Law:

$$n_1 \sin\theta_1 = n_2 \sin\theta_2. \quad (3)$$

Using Equation 3 and setting θ_2 equal to 90 degrees, the critical angle is found by

$$\sin\theta_c = \frac{n_2}{n_1}, \quad (4)$$

with θ_C as the critical angle. At any angle greater than θ_C, total internal reflection occurs, as seen in Figure 7 [8][10].

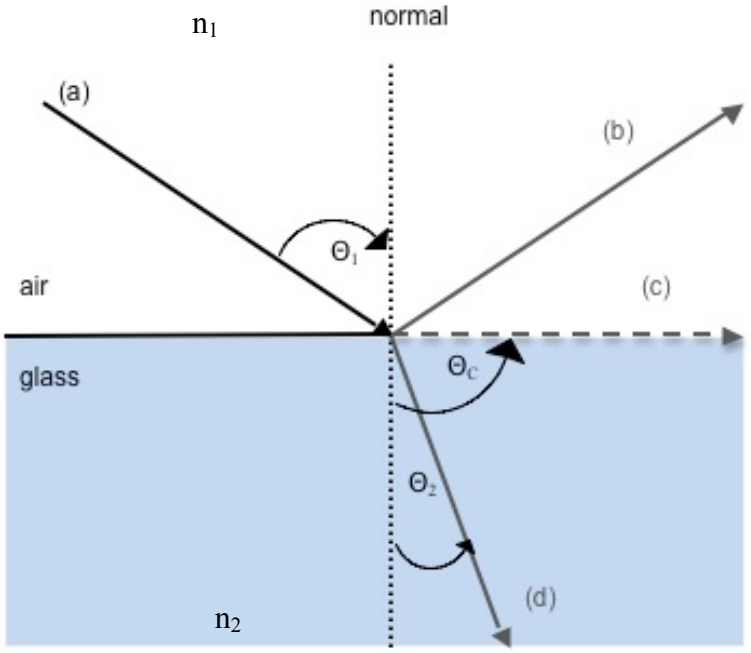


Figure 7. Depiction of a Light Source Incident Upon a Surface

(a): The incident ray of light; (b): reflected ray; (c): direction of light for total internal reflection; (d): refracted ray.

4.2 Evanescent Waves

When a laser light hits a glass prism, the light is reflected at the same angle as the angle of incidence. The light is reflected internally at angles greater than the critical angle and the photons of light tunnel through the gold slide in what is called an evanescent wave. The electric field created in the glass of the prism by this wave oscillates parallel to the surface of the material and exponentially decays perpendicular to the surface [10][11]. The wave that propagates along the surface adds to the electric field at the surface, as seen in Figure 8.

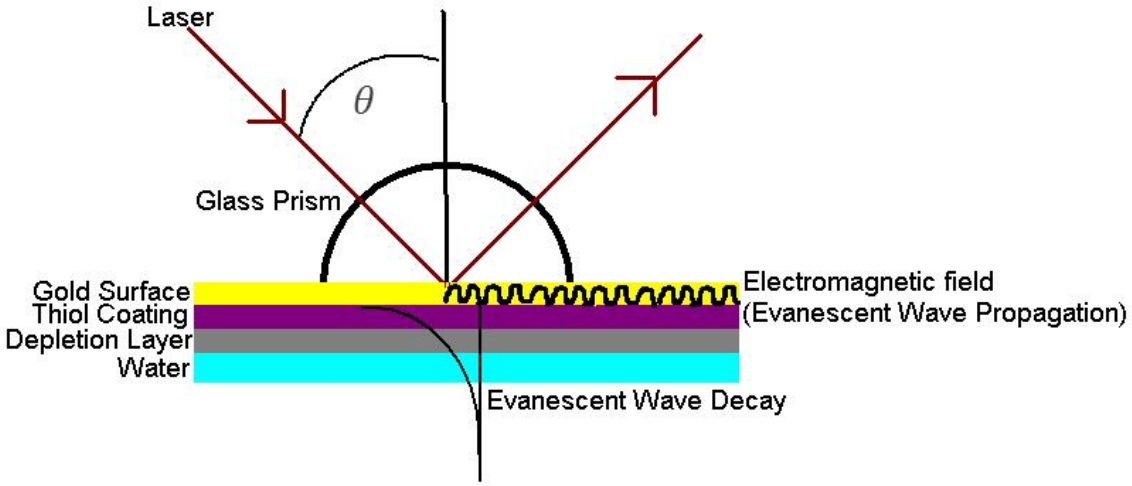


Figure 8. Diagram of a Generated Evanescent Wave

The image shows the incoming laser light forming an evanescent wave and its exponential decay at the reflective surface.

Image by Caitlyn Neidig.

4.3 Surface Plasmons

Surface plasmons are longitudinal charge density waves of free electrons that can be excited at an interface by an evanescent wave [12]. In order to excite surface plasmons, an induced surface charge density must be present. An induced surface charge density is created by a component of the electric field traveling in the direction perpendicular to the surface. The

helium-neon laser used in this experiment generates an electric field with components in both the parallel and the perpendicular directions in relation to the surface. The magnitude of the electric field is given by

$$E = E_0 e^{\frac{-z}{l}} e^{i(k_x x - \omega t)} \quad (5)$$

with $e^{\frac{-z}{l}}$ representing the decay of the evanescent wave, with z as the perpendicular distance to the surface and l as the decay length of the wave [11]. The decay length determines how far into the material the wave propagates. Here, l depends on the indices of refraction of the materials and the angle of incidence of the laser light and is given by

$$l = \frac{c}{\omega} \frac{1}{\sqrt{n_1^2 \sin^2 \theta_1 - n_2^2}} \quad (6)$$

where c is the speed of light and ω is the frequency of the evanescent wave. The second part of Equation 5,

$$e^{i(k_x x - \omega t)}, \quad (7)$$

represents the wave oscillations parallel to the surface of the material. The value k_x in Equation 7 is called the in-plane wavenumber and is given by [11]

$$k_x = \frac{\omega n_1}{c} \sin \theta_1. \quad (8)$$

4.4 Resonance and Technique

When the frequency of the evanescent wave and the frequency of the surface plasmons are the same, the resultant condition is called resonance. This occurs when the in-plane wave number (Equation 8) is equal to the propagation constant of the surface plasmons, K_x, in the equation below:

$$K_x = \frac{\omega^2}{c^2} \frac{\varepsilon_2 \left(1 - \frac{\omega_p^2}{\omega^2}\right)}{\left(\varepsilon_2 + 1 - \frac{\omega_p^2}{\omega^2}\right)}. \quad (9)$$

In Equation 9, ω_p is the plasmon frequency, ω is the angular frequency, and ε_2 is the dielectric coefficient of the sample [11]. The resonance condition then occurs when

$$k_x = K_x. \quad (10)$$

These concepts—total internal reflection, evanescent waves, surface plasmons, and resonance—are all important for the technique called surface plasmon resonance (SPR), which is used in order to study the optical thickness of the material layers. When an evanescent wave is created, energy is transferred from the exponentially decaying wave to the sinusoidally-propagating surface plasmon wave. This transfer of energy correlates to the dip in relative intensity of the transmitted laser beam at the resonance angle. The dip can be is viewed and measured using a computer program called LabVIEW.

5. Apparatus

Our lab uses SPR through a set-up comprised of a helium-neon laser with a wavelength of 632.8 nanometer, operating at a low, but very consistent intensity (power = 2.0 MW at 633 nm). The laser is aligned to shine through two collimating lenses and then travel through a cylindrical glass prism with index of refraction 1.723 and onto the gold-covered glass slide. The focal length of the first lens is 12.5 centimeters and the focal length of the second lens is 1.2 centimeters. This reduces the total width of the beam by a magnitude of 10. The collimation of the laser light is shown below in Figure 9.

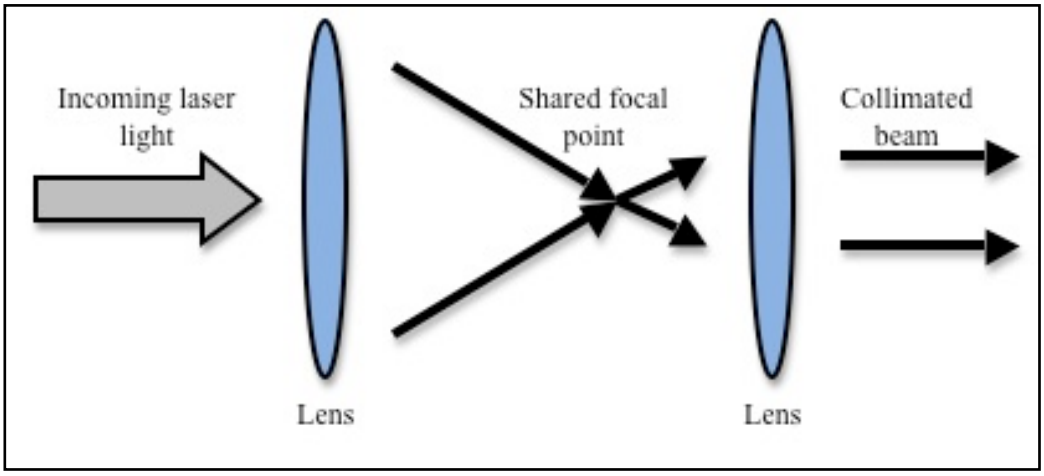


Figure 9. Collimation of The Laser Light Through Two Lenses
The beam travels through the two collimating lenses, which are lined up such that their focal points are the same. The beam is effectively narrowed to one-tenth its original height.

The gold of the slide, covered in a SAM of hydrophobic or hydrophilic molecules, releases the excited surface plasmons in the experiment. The gold slide fits into a Teflon block, which attaches to two Teflon screws into which two plastic tubes are inserted. The tubes are fitted tightly with Teflon tape, and the fluid is pumped into the sample cell area through these tubes. A rubber o-ring placed against the slide and fitted into the Teflon block secures the sample

cell. The ring helps to maintain a tight seal so that the fluid can be contained in the cell without leaking. Figure 10 shows a correctly set up sample cell.

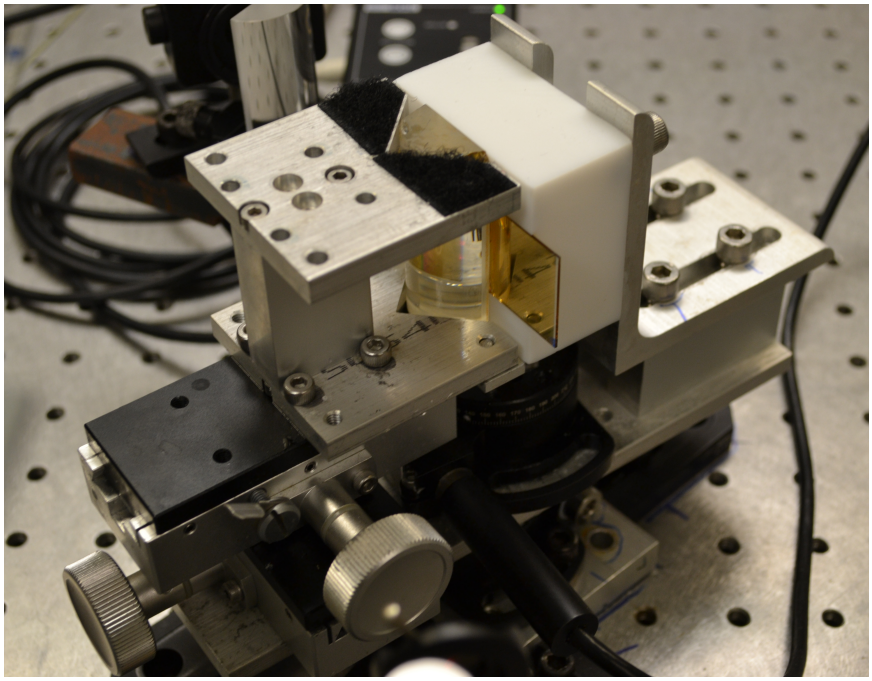


Figure 10. The Tight Sample Cell

This image above shows the tightly sealed sample cell, ready for a trial to be run.

The Teflon block, slide, and prism are mounted on top of two rotating stands on an optical lab table. The rotation stands are powered by two separate motors, allowing for independent adjustments of the angle of the sample in relation to the laser and the angle of the sample in relation to the photodetector. The detector measures the relative intensity of the incoming light. The mounting of the rotation stands directly below the sample cell ensures that the entire set-up rotates on the same axis. The laser light is directed into the detector via a second glass prism. By sweeping over a range of angles, a sampling of different wavevectors is achieved. When the wavevector reaches the value at the angle of resonance of the sample, the intensity of the light entering the detector drops, generating a dip in the graph of the intensity. A

diagram and an image of the complete experimental apparatus are given below in Figures 11 and 12, respectively.

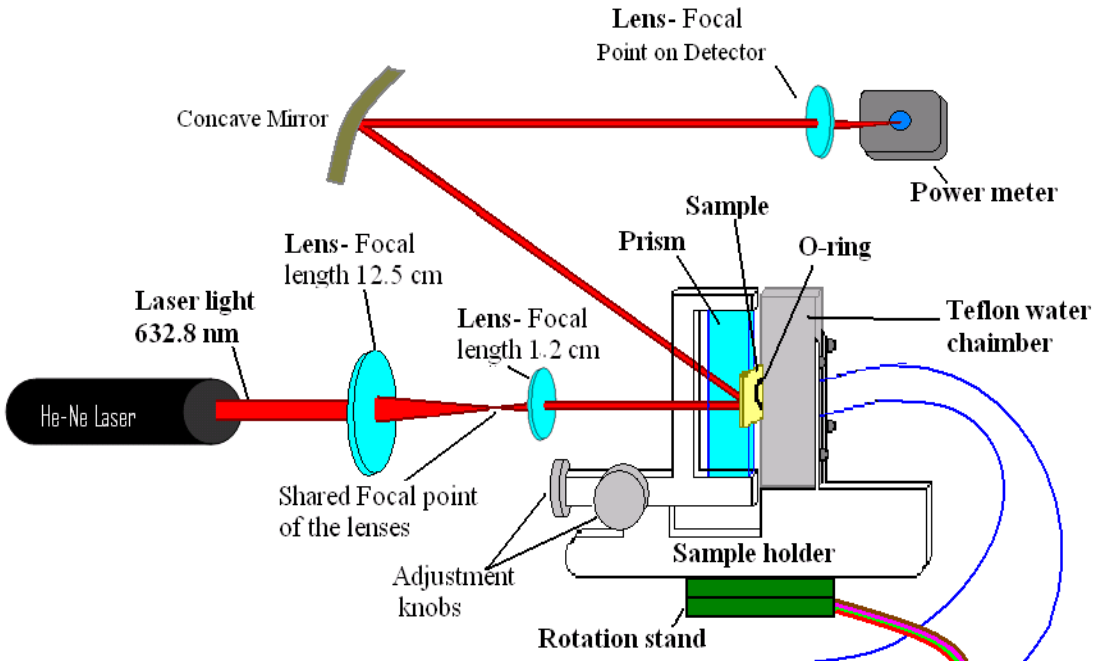


Figure 11. Diagram of the Complete Experimental Set-Up

This image shows a block diagram of the entire experimental apparatus. The helium-neon laser travels through the two collimating lenses to a prism and into the sample cell. The intensity of the outgoing beam is then measured by the photodetector.

Image by Poynor, Kohnstamm, and Chiang.

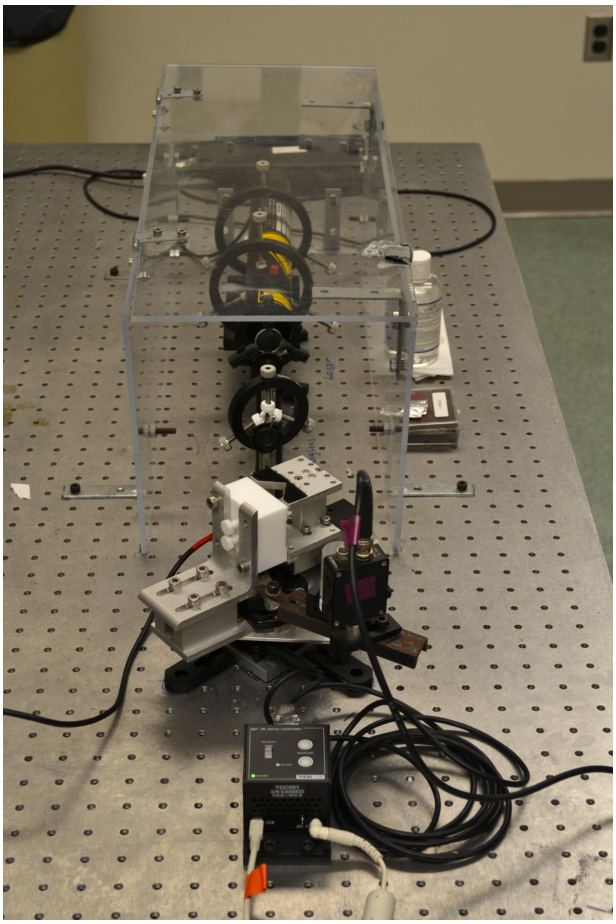


Figure 12. The Complete Experimental Set-Up

All of the components of the apparatus are lined up on the optical lab table.

SPR tests are run for air, water, and methanol and the resultant characteristics at the interface of water and either the hydrophobic or a hydrophilic surface are studied. Confining the test to a small sample cell, we can look at the region immediately next to the gold slide. Methanol is used in addition to air and water because it has optical properties similar to water but lacks hydrogen bonding so it does not form the low-density depletion layer at the surface. The results from the air trials are used as a baseline measurement with which to compare the other results.

6. Methods

6.1 Aligning the Laser

It is important for the laser light to converge completely in the sample cell so that an accurate reading can be made. In order for this to occur, the laser must be aligned correctly and collimated through the two lenses. The first lens is a convex-concave lens and the second lens is a concave-convex lens. When the focal points are lined up, the beam is collimated properly at the sample cell. The alignment is done with the assistance of two iris stands that can be opened and closed to different aperture sizes. The alignment process is shown in Figure 13.

One iris is placed close to the laser, and the other is placed farther away, past where the sample cell would be set up. The position of the laser is adjusted by moving the six screws holding it in place so that the beam travels though both the first iris and then the second.

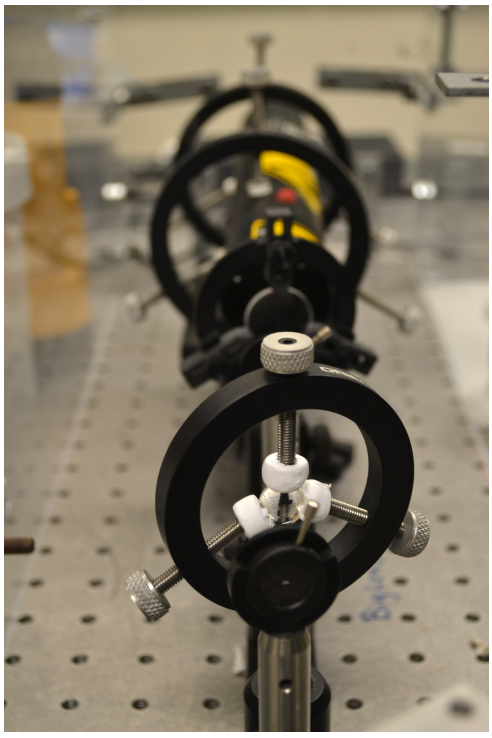


Figure 13. The Correct Alignment of the Laser

The laser beam is focused through the convex-concave and concave-convex lenses by using two iris stands, before reaching the sample cell.

6.2 Tight Sample Cell

Once the laser is aligned, the o-ring is placed against the gold side of the coated slide and the slide is fitted into grooves in the Teflon block. The slide is then sandwiched in place by the glass prism. To make certain there is the closest possible contact, the gold slide is coated with a thin layer of glycerin before the prism is brought into contact to eliminate air bubbles between the layers. Glycerin is used because it has approximately the same refractive index as the glass prism, around n = 1.5. Behind the slide is the sample cell, which measures three centimeters in diameter and one centimeter in depth. To prevent any movement during the trial, a screw tightens the slide to the Teflon block and a latch is locked. The sample cell then has a tight seal so that it can be left dry or filled with water or methanol without leakage.

6.3 Centering the Sample

Once the sample cell is set up tightly, it then has to be centered with the laser beam. This is done using a protractor attached to the top of the Teflon block, as pictured below (see Figure 14). The angle at which the laser light enters the prism and sample cell must be the same as the angle at which the light leaves the sample cell and travels to the detector. The beam is checked for at least three different angles, usually 20 degrees, 40 degrees, and 60 degrees. Small adjustments can be made to the position of the sample cell by using a knob on the side of the stand.

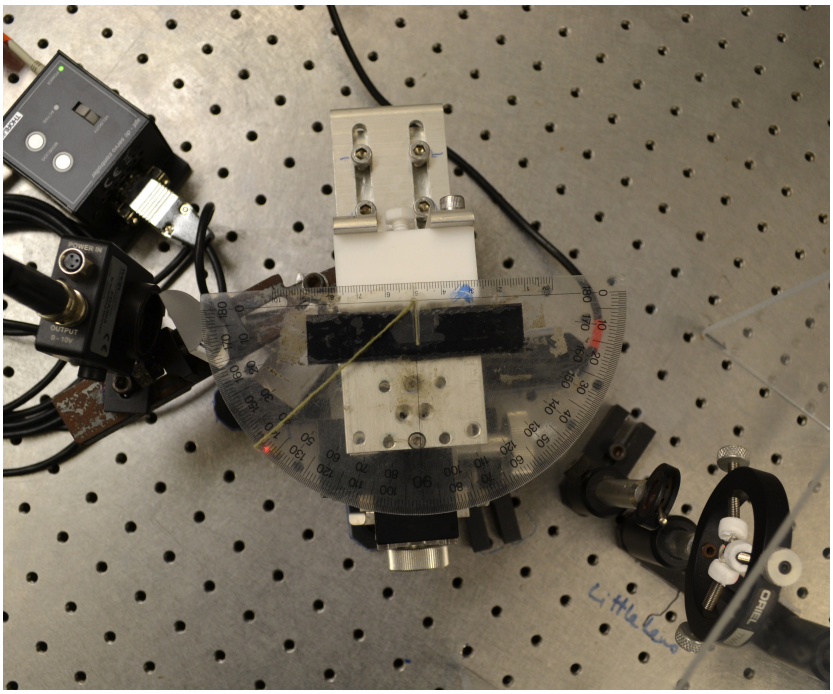


Figure 14. Centering of the Sample Cell

A protractor is lined up with the edge of the gold slide to make sure light enters and leaves the sample cell at the same angle.

6.4 Calibration Constant

In order to find the dip in intensity and thus the resonance angle, the sample is rotated in increments to cover a range of different angles. However, the angle of the sample and the angle of the photodetector arm do not match up exactly. The two parts need to be calibrated so that they rotate at the same rate. This calibration is done manually by placing the sample stand at a starting angle and rotating it in intervals of two degrees while matching up the detector arm with the laser beam exiting the sample cell at each step so that the beam goes into the detector at all angles. The process is completed over the whole range of angles to be studied to ensure the beam does not diverge at any location. The angles of the sample and the detector arm are recorded and then graphed on a computer, as shown in the Figure 15. The slope of the resultant line is the calibration constant.

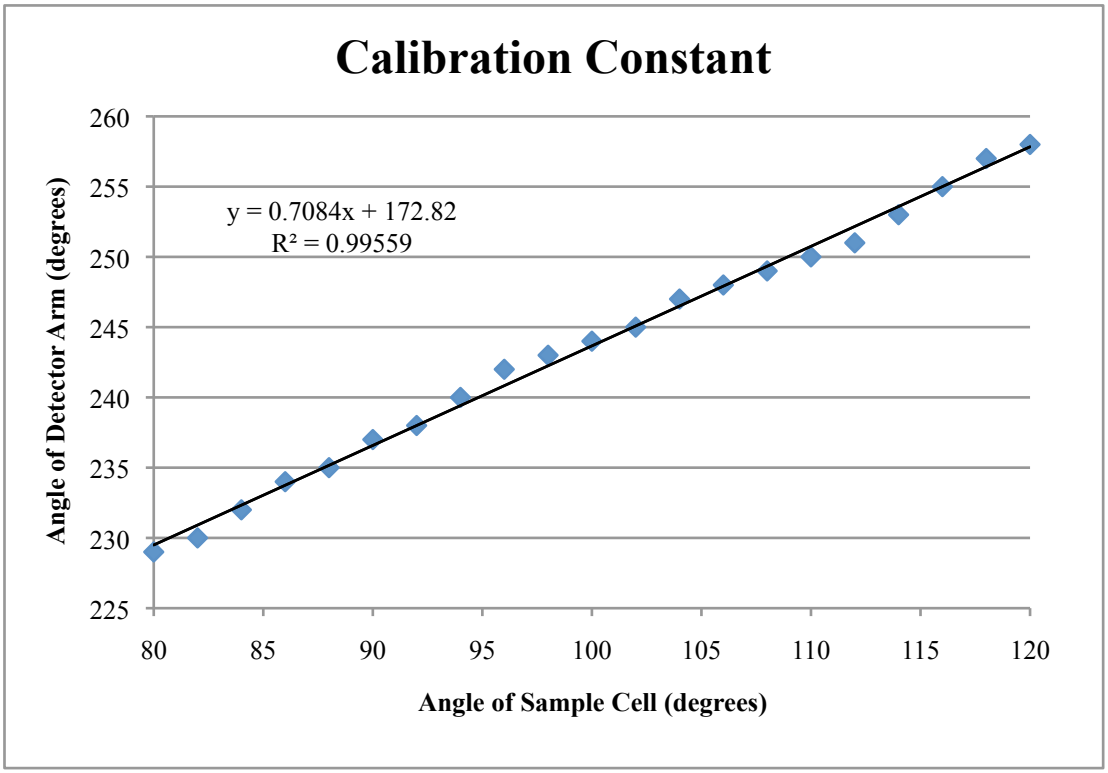


Figure 15. Determination of the Calibration Constant

The graph above shows the angle of the sample versus the angle of the detector. The slope represents the calibration constant, which regulates the rate at which different parts the apparatus rotate. The apparatus must be recalibrated before trials for each different slide.

6.5 Solution Making

To create the thiol slide coatings for the gold slides, stock solutions of ODT and MUA with concentrations of 0.1 M are made by adding 0.1 moles of the desired organothiol to 100 milliliters of ethanol volumetrically. These stock solutions can be stored and later diluted to the necessary concentration. For example, a one-millimolar solution must be diluted by a factor of 100, i.e. one milliliter of the one molar stock solution should be added to a 100-milliliter volumetric flask and filled to the mark with ethanol. After making either the stock solution or a particular dilution, the solution should be mixed thoroughly by repeatedly inverting the tightly sealed flask and ultrasonicated for 15 to 30 minutes or until completely dissolved. The solution is

ultrasonicated in order to remove any additional organothiol molecules from the SAM. If particles are still visible, the solution should continue to be mixed and then allowed to sit for a period of time until no particulate remains. After the organothiol is totally dissolved, the solution can be used for slide production. For this experiment, one-millimolar solutions were used in coating each slide.

6.6 Slide Production

Once the solutions are made, a clean bare gold slide can be coated with a SAM of the desired organothiol by placing it in a skinny beaker and adding enough of the appropriate solution to completely cover the slide. The beaker is covered in foil and left to sit overnight. The slide is then rinsed five times in ethanol, placed in a clean beaker, and submersed in ethanol. The beaker containing the slide and ethanol is covered in foil and placed in a larger beaker filled with water. This beaker is covered with foil as well and ultrasonicated for ten minutes. Finally, the slide is rinsed with ethanol five times again and placed in a clean beaker, covered in foil, and left to dry (see Figure 16). In order to achieve the full range of contact angles, different mixtures of ODT and MUA were used. Possible mixtures were first tested on small individual slide pieces. If the desired contact angle was found, then a full slide was coated.



Figure 16. An Organothiol-Coated Slide Drying in a Beaker
This gold slide is placed in a covered skinny beaker to dry after being coated with the organothiol and rinsed five times in ethanol.

6.7 Calculating Contact Angles

To find the contact angle, the slide is placed with the gold side up on a sheet of lens paper on the platform of the goniometer, as shown in Figure 17. Several drops of water are placed along the slide (see Figure 18). Crossbars within the goniometer are lined up with the surface of the slide and then the scope is rotated to line up with the edge of the drop in order to measure the angle. This process is repeated several times for each drop of water and for several different drops on the surface. Then the angles are averaged to find the most accurate contact angle and thus the most accurate reading of the degree of hydrophobicity. Contact angles can also be measured using a camera hooked up to the computer (see Appendix 10.3).

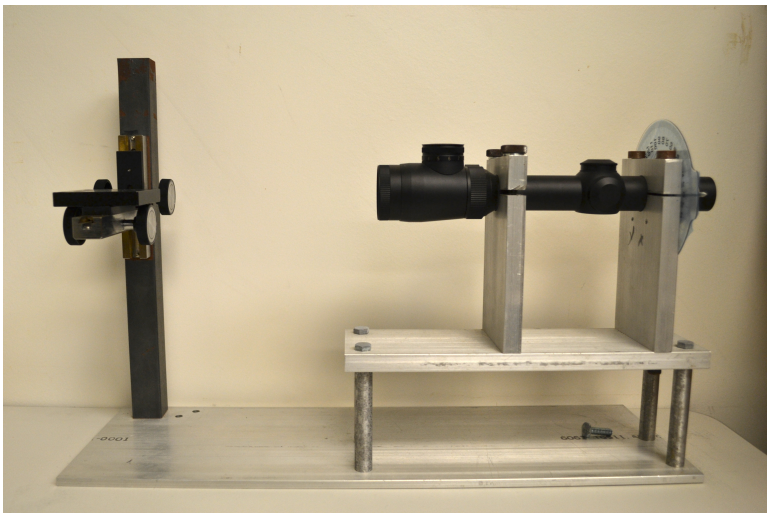


Figure 17. Goniometer Used to Measure Contact Angles

The goniometer can be used to measure the contact angle of the water drops on the slide surfaces.

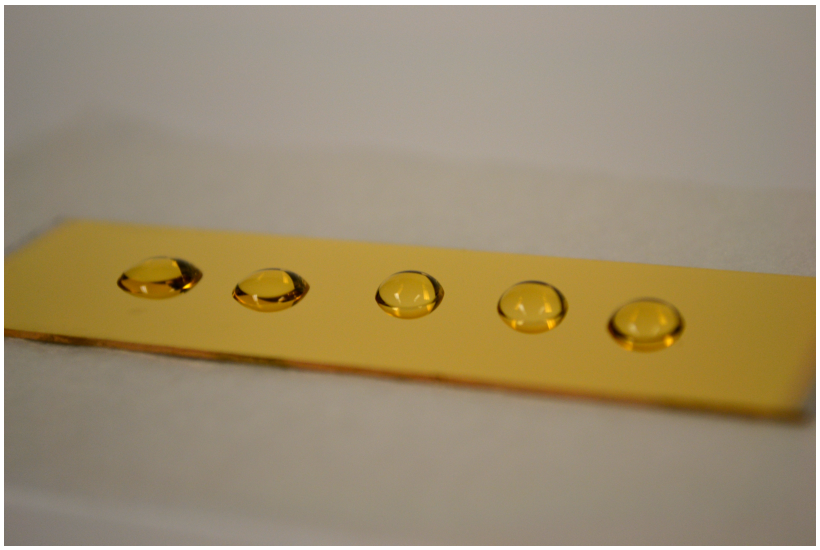


Figure 18. Water Drops on the Gold Slide

Several drops are placed on each slide so that an average of the measured contact angles can be used.

6.8 LabVIEW

The determined calibration constant, along with the starting and ending angles of rotation for the sample within the desired range, and the desired interval are all plugged into a computer

program called LabVIEW. LabVIEW tracks the progress of the data collection, recording the relative intensity of the laser in the detector at each different angle for the sample stand within the range. A screen-shot of the main panel of the program is shown below in Figure 19.

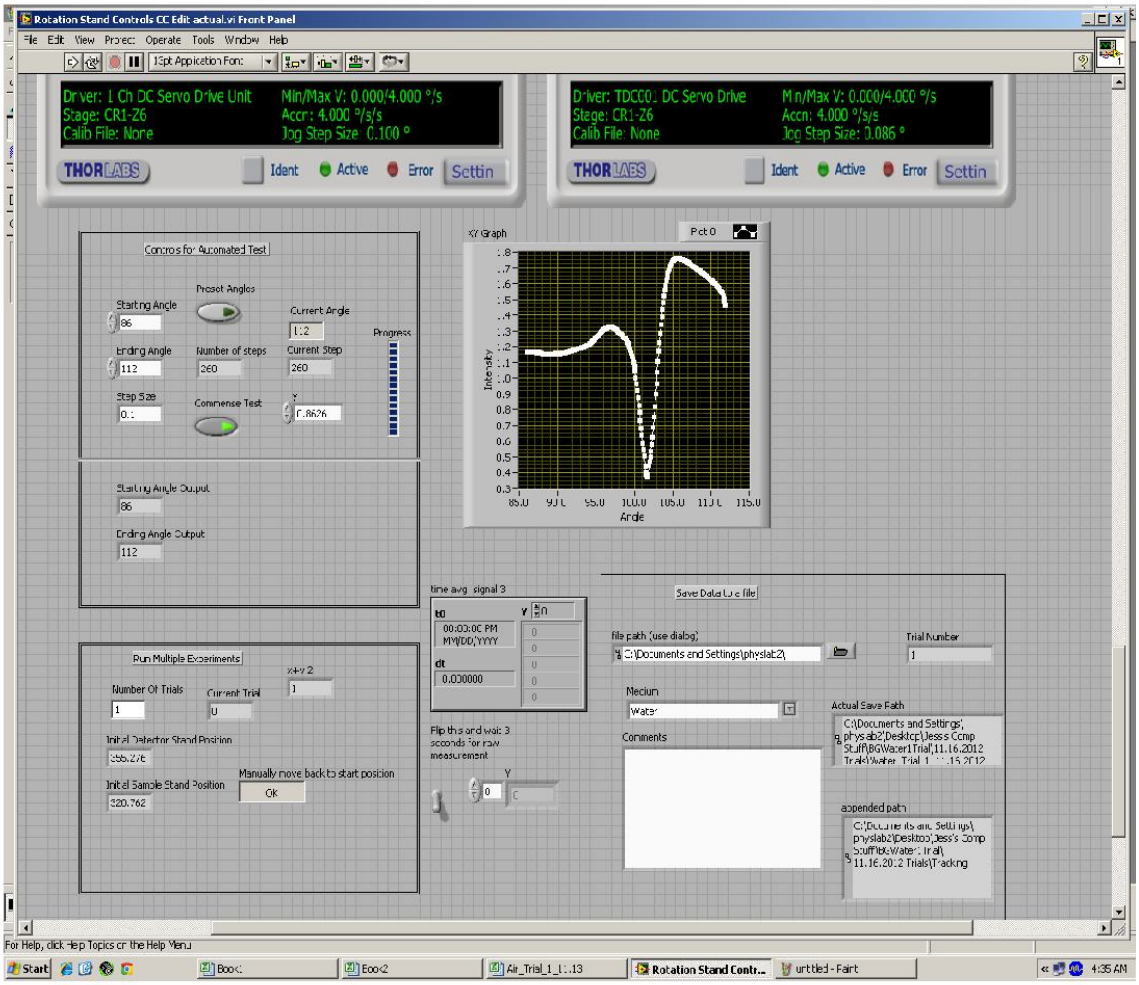


Figure 19. Main Screen of LabVIEW

The top panels of the main screen of the program are the motor controls and the bottom half of the screen shows the data being collected from the current trial.

6.9 SPRangle Data

The SPRangle computer program generates plots of theoretical data for each experimental trial, allowing a comparison to be made between the experimental results from the

LabVIEW program and the generated theoretical results. Information about the types of surfaces, specifically their thicknesses and indices of refraction, must be entered. The indices of refraction used are in Table 2 below, and a sample input for the SPRangle program is pictured as well (Figure 20).

Material	n (real)	n (imaginary)	Thickness (angstroms)
Air	1.00	0	-
Depletion	1.00	0	20-50
Glass	1.732	0	-
Gold	0.1726	3.422	500
Methanol	1.328	0	-
MUA	1.483	0	20
ODT	1.45	0	20
Water	1.33	0	-

Table 2. SPRangle Input Values for Various Materials

This table contains the refractive indices and thicknesses for various materials used in the experiment. These values are necessary to generate theoretical results to which the experimental data is compared.

```
BGAirP

3         :num_phase (number of phases)
6328      :lambda (wavelength)
0         :begtheta (starting angle)
120       :endtheta (ending theta)
.1        :inctheta (angle increment)

1.732     :n[1].rl (real part of n1)          Prism
0         :n[1].im (imaginary part of n1)

0.1726    :n[2].rl (real part of n2)          Gold
3.422     :n[2].im (imaginary part of n2)
500       :d[2] (Thickness of phase 2)

1.0       :n[3].rl (real part of n3)          Medium
0         :n[3].im (imaginary part of n3)
```

Figure 20. Actual SPRangle Input

The text above is the input for a bare gold air trial. The input varies slightly for different trials with different slides. For full input, see Appendix 10.6.

7. Results and Discussion

7.1 Bare Gold Slide

The trial runs were carried out for each of the slides and the results are graphed below. In each figure, the dip in the graph represents the angle at which the resonance condition occurs. The small maxima before the dips represent when the critical angle is reached and total internal reflection begins. The intensity is scaled such that the highest recorded intensity is equal to one. For each angle in the water and methanol trials, the resonance angle for the air trial is subtracted so that the values for the various trials are compared to the same starting point. For details on how the data is analyzed, see Appendix 10.7. Figure 21 shows the results for the bare gold slide, which is used as a comparison to the coated slides. A difference of 0.6 degrees was found between the theoretical resonance angle and the experimental resonance angle; this value represents systematic error.

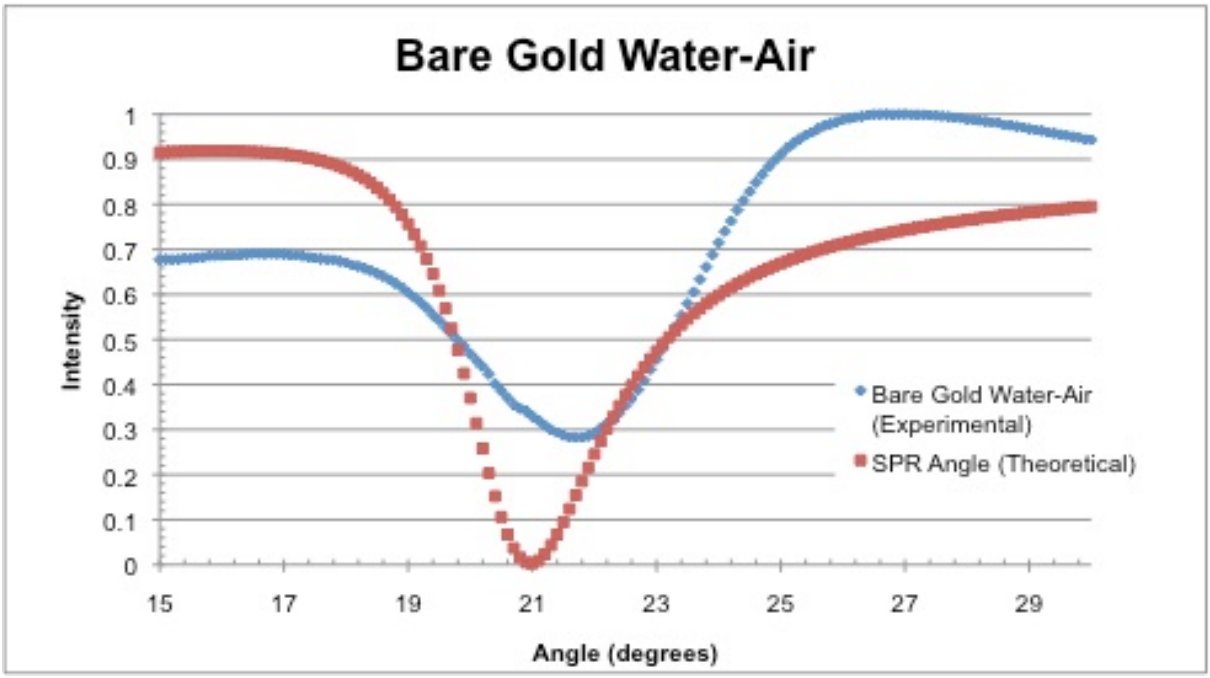


Figure 21. Results for the Bare Gold Slide

The graph above shows the theoretical graph generated by SPRangle for the bare gold slide. The contact angle of the bare slide was between 78 and 79 degrees.

7.2 30° Contact Angle Slide

Figure 22 shows the results from the slide coated in pure MUA. The different curves represent the data from the methanol trials, the water trials, the theoretical methanol data, and the theoretical water data for various depletion layers. The correctly shifted curve shows the presence of a 30-angstrom thick depletion layer. The MUA slide has a contact angle of 30.0 degrees, the lowest of any slide tested, and one would therefore expected the slide to correlate to the thinnest depletion layer.

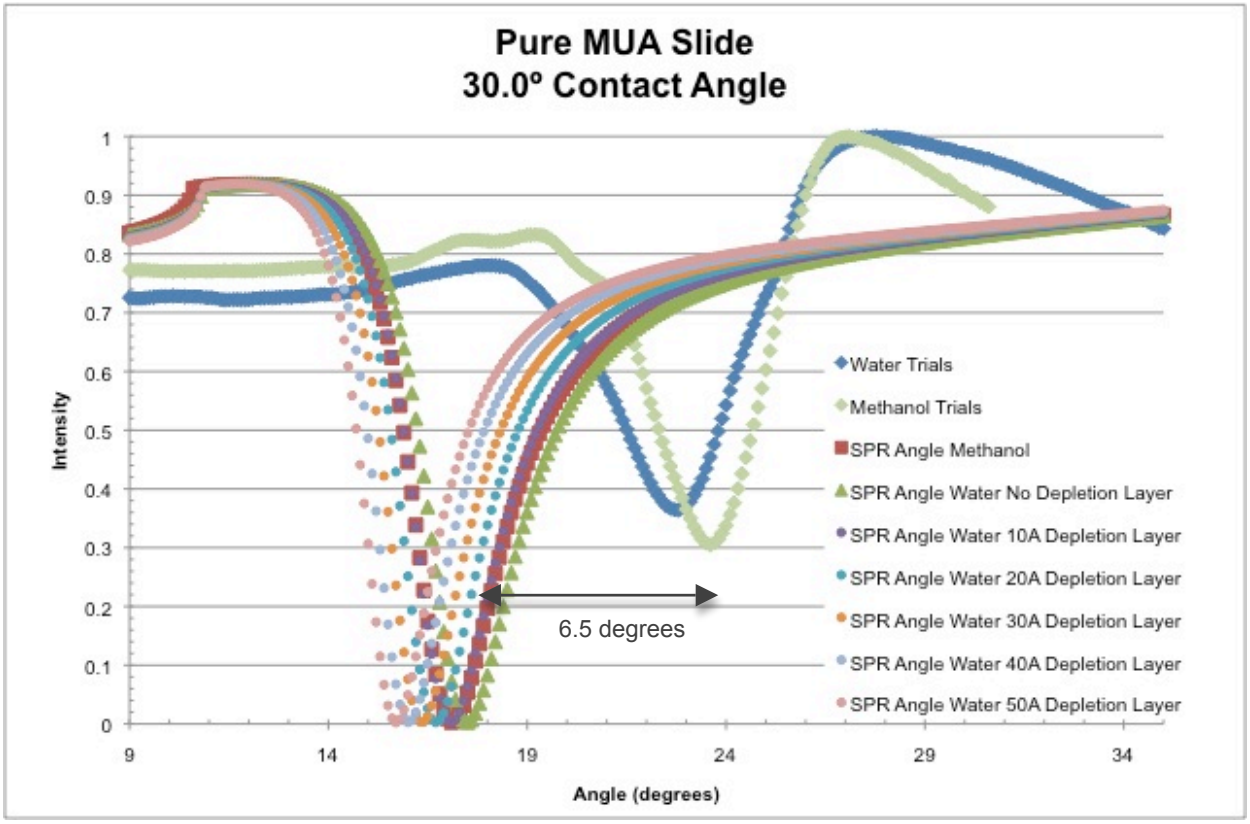


Figure 22. Results for the Pure MUA-Coated Slide

The graph shows the results from the MUA slide, which has a contact angle of 30.0 degrees. The difference between the experimental and theoretical resonance angles was 6.5 degrees, which corresponds to the theoretical difference between the water trails and the curve for the 30-angstrom depletion layer.

7.3 61.9° Contact Angle Slide

Then trials were run for slides coated in mixed solution. The results from the trials for the 20% ODT: 80% MUA slide are shown below in Figure 23. The difference between the experimental and theoretical methanol trials was found to be 0.8 degrees. When compared to the water trials, this difference corresponds to the curve for no depletion layer. Given that the MUA slide with a smaller contact angle shows a 30-angstrom depletion layer, this is an unexpected result. It is predicted that for larger contact angles, the depletion layer would increase in thickness.

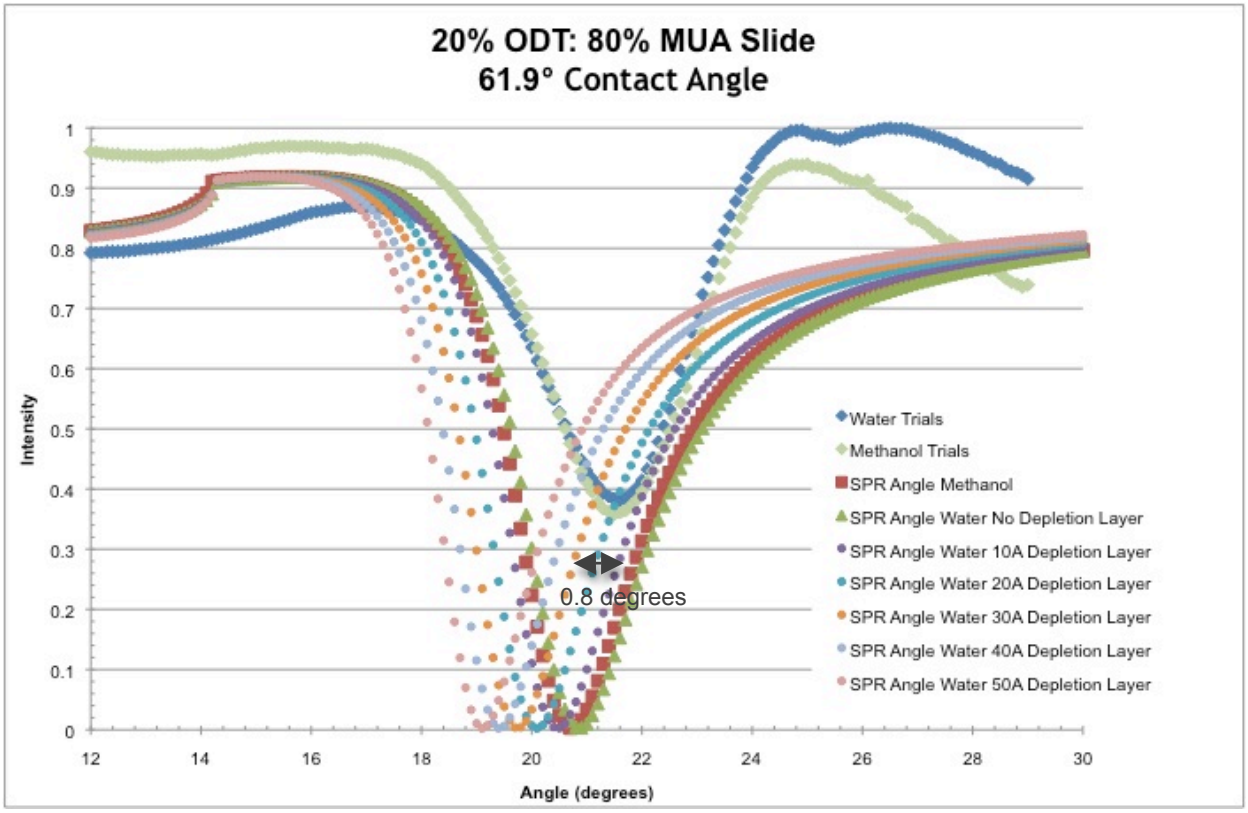


Figure 23. Results for the 20% ODT: 80% MUA Slide with 61.9° Contact Angle

The graph shows the results from the 20% ODT: 80% MUA slide, which has a contact angle of just under 62 degrees. The difference between the methanol experimental and theoretical dip angles was 0.8 degrees, which corresponds to the difference between the experimental water trials and the curve for no depletion layer.

7.4 79.9° Contact Angle Slide

The results from the trials for the 35% ODT: 65% MUA slide with a contact angle of 79.9 degrees are given in Figure 24 below. The difference in dip angles corresponds to the 10-angstrom depletion layer for the slide in contact with water. This accuracy of this thickness is not known. In comparison to the 61.9° slide, a 10-angstrom depletion layer seems plausible, but when compared to the 30.0° slide, one would expect the thickness for the 35% ODT: 65% MUA slide to be greater.

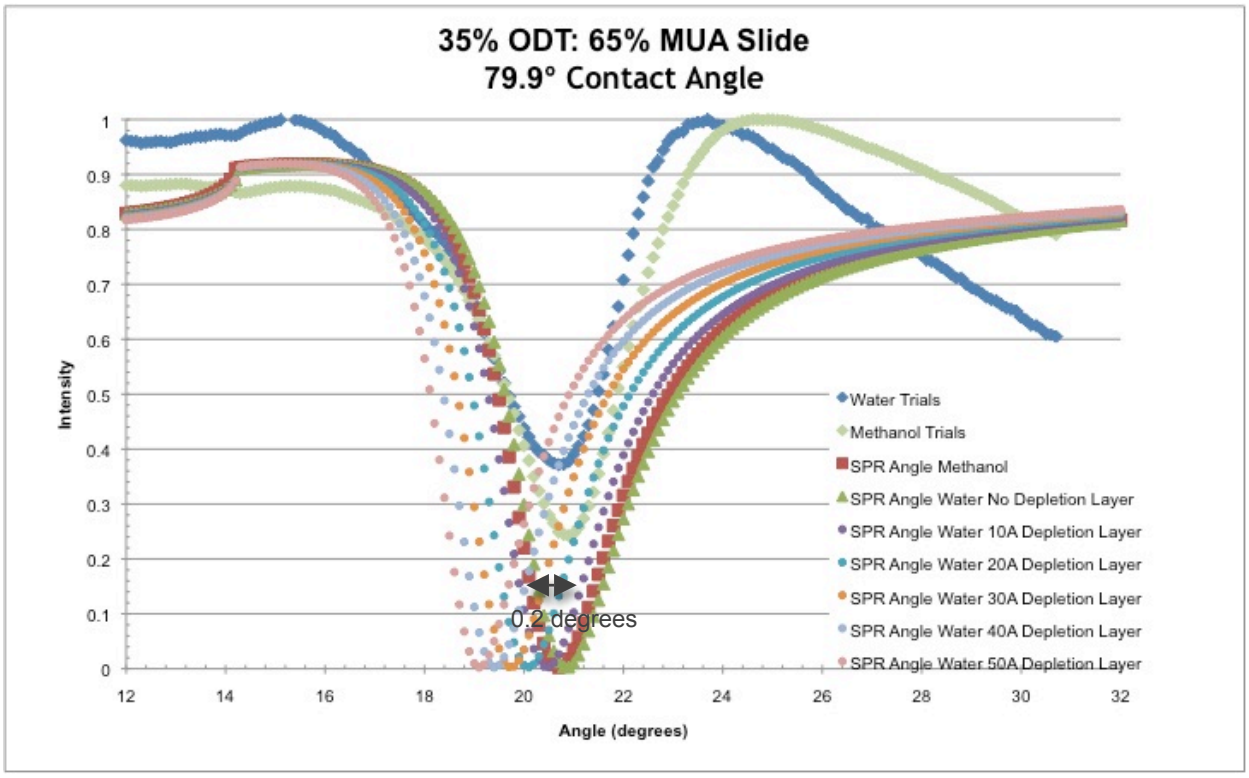


Figure 24. Results for the 35% ODT: 65% MUA Slide with 79.9° Contact Angle

The graph shows the results from the 35% ODT: 65% MUA slide, which has a contact angle of 79.9° degrees. The difference between the methanol resonance angle and the experimental angle was 0.2 degrees, which corresponds to the theoretical difference between the water trails and the curve for the 10-angstrom depletion layer.

7.5 102.2° Contact Angle Slide

Figure 25 shows the results from the trials for the pure ODT-coated slide. The graph shows that the shift in curves indicates the slide has no depletion layer, which is unlikely considering ODT is a very hydrophobic solution and should therefore correspond to the thickest depletion layer.

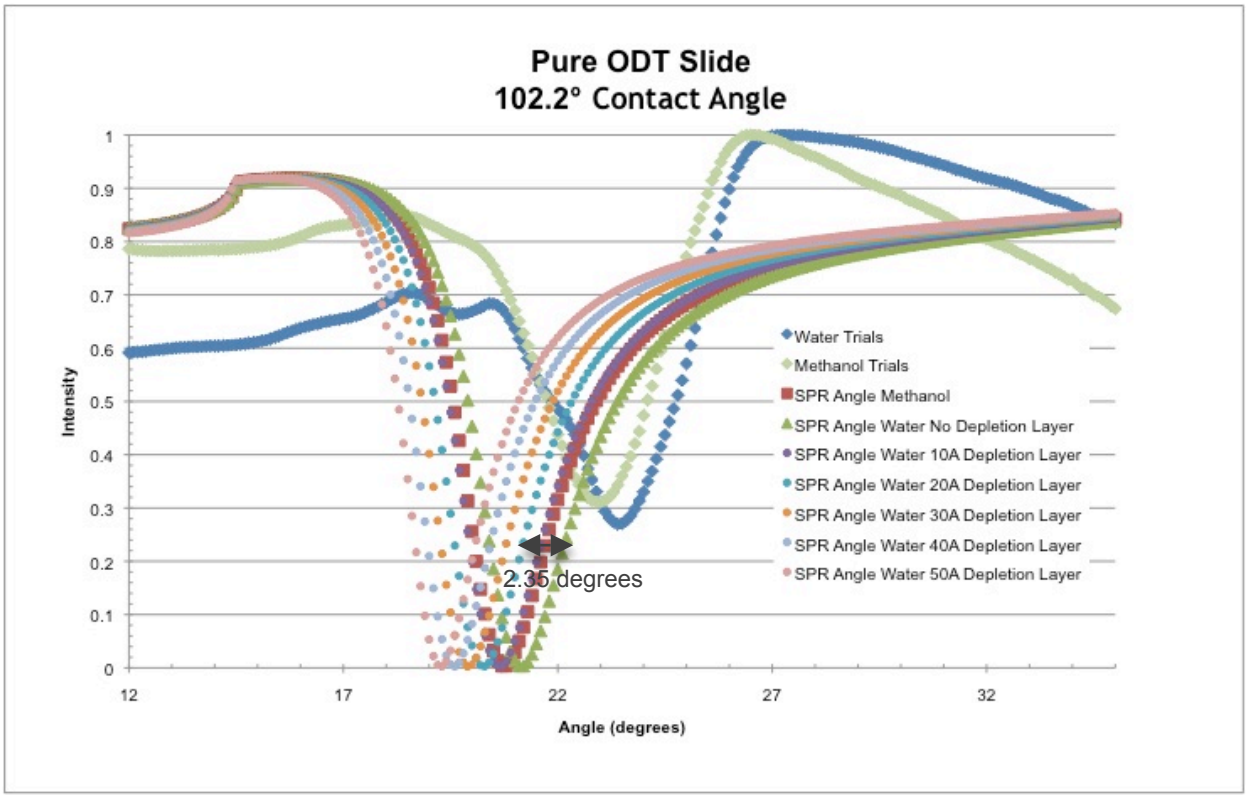


Figure 25. Results for the Pure ODT-Coated Slide with 102.2° Contact Angle

The graph above shows results from the ODT slide, which has a contact angle of 102.2 degrees. The difference between the methanol resonance angle and the experimental angle was 2.35 degrees, which corresponds to the theoretical difference between the water trials and the curve for no depletion layer.

7.7 Effect of Hydrophobicity on Depletion Layer

Figure 26 shows the combined results from all the slides. By comparing the experimental and theoretical data for both the water and the methanol trials, scaled by the results for the air trials, the thickness of the depletion layer is determined. The thicknesses are shown in

comparison to the contact angle of the slide. The graph includes the data points from the experiment by Neidig as well.

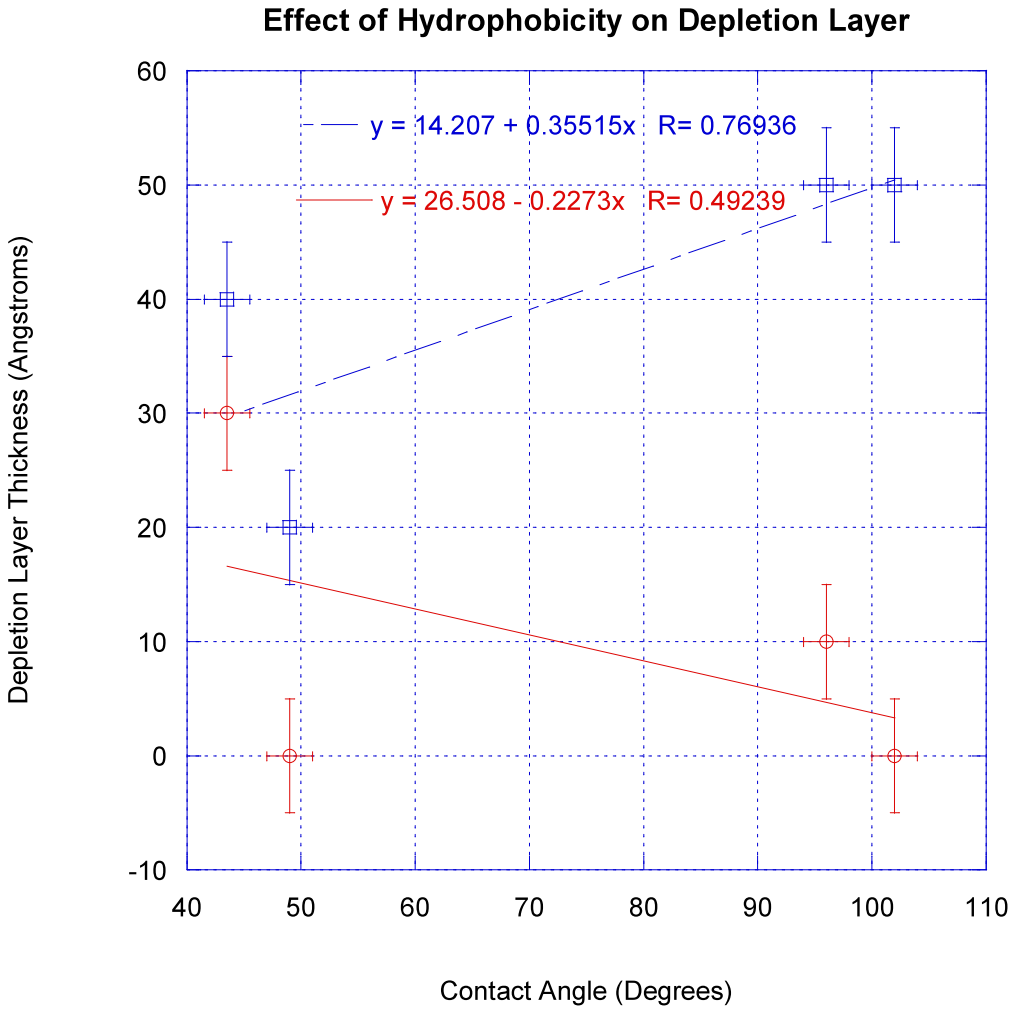


Figure 26. Graph of Effect of Hydrophobicity on Depletion Layer

The red data points are from this experiment and the blue points are from the experiment by Neidig. The vertical error bars show the error within the depletion layer thickness and the horizontal error bars show the determined error from measuring contact angles with the computer.

The figure shows that the results did not follow the expected trend. Data from this experiment does not agree with the results from previous experiments. Reasons for this

discrepancy are most likely due to differences in the roughness of the coating on the slides and variations in stability. The two slides coated in mixed solutions would have SAMs consisted of carbon chains of two different lengths. ODT has a chain length of 11 carbons and MUA has a chain of 18 carbons. When these two organothiols form a single solution, the result is a monolayer with a slight variation in thickness, depending on the exact placement of the ODT and MUA. This variation in thickness, or roughness, can affect the way water or methanol interacts with the surface. Longer chain lengths could dominate in the mixture or uneven distribution of the two different molecules could lead to skewed variations in the interaction.

8. Future Work

In the future, other students can continue to produce slides with different contact angles to expand the range of angles covered and determine the relationship between contact angle and the depletion layer for surfaces of varying degrees of hydrophobicity. However, the quality of the SAM, especially the stability of the layer, should first be studied. By measuring the contact angle of a single slide over a period of time, the stability of the slide can be determined by whether or not there is a change in the measurement. If a stable monolayer can be established, the results will be more reliable. A better understanding of how the roughness of mixed solutions affects the interaction may lead to further information about the how water interacts with surfaces of varying degrees of hydrophobicity as well. The roughness of the coatings can be determined by other methods, such as atomic force microscopy. Different organothiol mixtures, especially those with very similar chain lengths could produce a potentially more accurate picture of the phenomena occurring at a hydrophobic surface.

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10. Appendices

10.1 Making and Disposing of Baths

New Potassium hydroxide (KOH) baths should be made at the beginning of the school year and periodically throughout the year as needed. It is best to have more than one bath prepared at a time so that more things can be cleaned at once.

To make a bath, use 1 cup of KOH pellets in a container and fill the container two-thirds of the way full with isopropyl alcohol (use vinegar in small amounts to neutralize). Allow the new bath to sit for 12 hours before using. Baths start out clear and turn darker brown as they get older.

Always pour waste chemicals into a labeled waste container (include the name of all chemicals present). When the waste container is full or a bath is to be disposed of, fill out a waste removal sticker (neon yellow, in the top drawer) completely and notify the person who removes chemicals from labs on campus.

10.2 Cleaning Glassware

Everything in the lab must be cleaned regularly and kept free of dust at all times. Make a cover for the sample cell to keep dust off of it when not in use. All glassware is placed in the KOH bath in the fume hood. Make sure the glass is completely submerged in the bath and leave it to soak for at least 14 hours. Always wear a lab coat, long pants, closed-toe shoes, goggles, latex and food gloves when working with the bath.

To clean flasks, funnel KOH into them (it is more important to clean the inside than the outside) and cover the top in foil (do not use stopper).

After 14 hours, rinse glassware at least five times with ultrapure DI water (from the spectroscopy lab across the hall). Rinsing more than five times may be necessary. Use a syringe to pump water through plastic tubes.

Bake all the items in the toaster oven for 30 minutes at 300°F. Keep everything wrapped in foil when not in use/transporting to prevent exposure to dust.

Put screws and o-rings in a small beaker in the bath so they do not get lost. The o-rings do not go in oven; they are just placed in foil immediately. Double-check the holes in the Teflon block and screws for residue when rinsing. Clean the Teflon block, screws, and o-ring again before running actual trials. Anything spilled in the fume hood should be neutralized with baking soda, and then the baking soda should be cleaned up as well. Never screw the lid of the bath container on too tightly or it will not come off again.

10.3 Using the Computer to Determine Contact Angles

On the old computer, the username and password are both "Chem Admin."

To use the contact angle program:

1. Adjust so the entire drop fits on screen.
2. Adjust tilt and fit the blue box around the drop with the bottom corners at contact points.
3. Hit "rec", then "next."
4. Have the "all" option marked, then press "fit."
5. This should give you a contact angle but you may need to adjust box or restart program.
6. Repeat for as many angles as you want.

On new computer, adjust the stand and the slide until the drop is clearly visible on the computer screen, and then print the image. Measure the contact angle by hand with a ruler and a

protractor. Work quickly and efficiently because the contact angle can drop over time due to poor stability.

10.4 Cleaning Lenses and Prisms

Clean both prisms before starting actual trials. Use Kodak Professional Lens Cleaner (or the equivalent) and lens cleaning sheets. Always wear food gloves and try to handle the glass as little as possible.

Put one or two drops of solution on the prism, place the lens sheet on top, and pull the edge of the sheet to spread the solution across the surface. Do not rub or apply pressure to the sheet on the prism—it will leave scratches. Clean both sides of prism. There are some scratches on the glass but the prisms should appear clean.

Keep the prisms in their designated boxes when not in use. Do not drop or scratch the prisms.

Clean the lenses in their stands if possible. Clean the lenses in the same way you would clean the prisms, with Kodak Cleaning solution and lens paper. Do not apply direct pressure to the surface and always wear food gloves.

If the lenses are removed from the stands, make sure they are put back in securely and lined up straight so that the laser light travels correctly.

10.5 APT and LabVIEW (modified from Isaac Marx's Senior Research Project)

Before running any trials, make sure everything is plugged in. Open APT Config on the desktop to ensure that the computer recognizes both motors. Once both motors are recognized,

close APT Config and open LabVIEW on the desktop. (Do not try to have both programs open at once.) Open the correct version of the file titled “Rotation Stand Controls.”

Enter the starting angle, the ending angle, the step size, the calibration constant, and the number of trials. Select air, water, or methanol from the drop-down list and select a location in which to save the data. Make sure the range of angles includes the angle at which the dip is expected to occur. Start with trials running in 0.5 degree increments to make sure the calibration is correct. Then change the increment to 0.1 degrees and run actual trials. Make sure everything is running correctly for a few trials before leaving the program to run multiple consecutive trials. If necessary, run only one trial at a time.

To run trial, click the arrow marked “Run” and wait for two error messages to come up. Cancel both of the errors and then click the stop sign button. Click “Commence Test” and then click “Run” again to begin data collection.

Make sure to manually reset the sample cell and detector to the starting angles before beginning a series of trials. Keep the lights off during trials and put the “Do Not Disturb” sign on the lab door. After the trials are complete, click the stop sign button again. Close out of LabVIEW and check to make sure the trial data saved correctly.

10.6 SPRangle Input and Use

Open the sprangle folder on the desktop. Open the file called “a.txt” and enter the appropriate information into each section of the program. Change the number of phases (or layers), indices of refraction, and thicknesses depending on which trial data is being generated.

Sample SPR input for each type of trial is given below.

BGAir

```
3       :num_phase (number of phases)
6328    :lambda (wavelength)
0       :begtheta (starting angle)
120     :endtheta (ending theta)
.1      :inctheta (angle increment)

1.732   :n[1].rl (real part of n1)              Prism
0       :n[1].im (imaginary part of n1)

0.1726  :n[2].rl (real part of n2)              Gold
3.422   :n[2].im (imaginary part of n2)
500     :d[2] (Thickness of phase 2)

1.0     :n[5].rl (real part of n5)              Medium
0       :n[5].im (imaginary part of n5)
```

BGWater

```
3       :num_phase (number of phases)
6328    :lambda (wavelength)
0       :begtheta (starting angle)
120     :endtheta (ending theta)
.1      :inctheta (angle increment)

1.732   :n[1].rl (real part of n1)              Prism
0       :n[1].im (imaginary part of n1)

0.1726  :n[2].rl (real part of n2)              Gold
3.422   :n[2].im (imaginary part of n2)
500     :d[2] (Thickness of phase 2)

1.333       :n[5].rl (real part of n5)          Medium
0           :n[5].im (imaginary part of n5)
```

CoatedAir

```
4       :num_phase (number of phases)
6328    :lambda (wavelength)
0       :begtheta (starting angle)
120     :endtheta (ending theta)
.1      :inctheta (angle increment)

1.732   :n[1].rl (real part of n1)              Prism
0       :n[1].im (imaginary part of n1)

0.1726  :n[2].rl (real part of n2)              Gold
3.422   :n[2].im (imaginary part of n2)
500     :d[2] (Thickness of phase 2)

1.47145 :n[3].rl (real part of n3)              Layer
0       :n[3].im (imaginary part of n3)
20      :d[3] (Thickness of phase 3)

1.00    :n[4].rl (real part of n4)              Medium
0       :n[4].im (imaginary part of n4)
```

CoatedMeth

```
4       :num_phase (number of phases)
6328    :lambda (wavelength)
0       :begtheta (starting angle)
120     :endtheta (ending theta)
.1      :inctheta (angle increment)

1.732   :n[1].rl (real part of n1)              Prism
0       :n[1].im (imaginary part of n1)

0.1726  :n[2].rl (real part of n2)              Gold
3.422   :n[2].im (imaginary part of n2)
500     :d[2] (Thickness of phase 2)

1.47145 :n[3].rl (real part of n3)              Layer
0       :n[3].im (imaginary part of n3)
20      :d[3] (Thickness of phase 3)

1.328   :n[4].rl (real part of n4)              Medium
0       :n[4].im (imaginary part of n4)
```

CWaterNoDep

```
4       :num_phase (number of phases)
6328    :lambda (wavelength)
0       :begtheta (starting angle)
120     :endtheta (ending theta)
.1      :inctheta (angle increment)

1.732   :n[1].rl (real part of n1)          Prism
0       :n[1].im (imaginary part of n1)

0.1726  :n[2].rl (real part of n2)          Gold
3.422   :n[2].im (imaginary part of n2)
500     :d[2] (Thickness of phase 2)

1.47145 :n[3].rl (real part of n3)          Layer
0       :n[3].im (imaginary part of n3)
20      :d[3] (Thickness of phase 3)

1.333   :n[4].rl (real part of n4)          Medium
0       :n[4].im (imaginary part of n4)
```

CWater10Dep

```
5       :num_phase (number of phases)
6328    :lambda (wavelength)
0       :begtheta (starting angle)
120     :endtheta (ending theta)
.1      :inctheta (angle increment)

1.732   :n[1].rl (real part of n1)          Prism
0       :n[1].im (imaginary part of n1)

0.1726  :n[2].rl (real part of n2)          Gold
3.422   :n[2].im (imaginary part of n2)
500     :d[2] (Thickness of phase 2)

1.47145 :n[3].rl (real part of n3)          Layer
0       :n[3].im (imaginary part of n3)
20      :d[3] (Thickness of phase 3)

1.0     :n[4].rl (real part of n4)          Depletion Layer
0       :n[4].im (imaginary part of n4)
10      ;d[4] (Thickness of phase 4)
```

```
1.333       :n[5].rl (real part of n5)          Medium
0           :n[5].im (imaginary part of n5)
```

CWater20Dep

```
5       :num_phase (number of phases)
6328    :lambda (wavelength)
0       :begtheta (starting angle)
120     :endtheta (ending theta)
.1      :inctheta (angle increment)

1.732   :n[1].rl (real part of n1)          Prism
0       :n[1].im (imaginary part of n1)

0.1726  :n[2].rl (real part of n2)          Gold
3.422   :n[2].im (imaginary part of n2)
500     :d[2] (Thickness of phase 2)

1.47145 :n[3].rl (real part of n3)          Layer
0       :n[3].im (imaginary part of n3)
20      :d[3] (Thickness of phase 3)

1.0     :n[4].rl (real part of n4)          Depletion Layer
0       :n[4].im (imaginary part of n4)
20      ;d[4] (Thickness of phase 4)

1.333   :n[5].rl (real part of n5)          Medium
0       :n[5].im (imaginary part of n5)
```

CWater30Dep

```
5       :num_phase (number of phases)
6328    :lambda (wavelength)
0       :begtheta (starting angle)
120     :endtheta (ending theta)
.1      :inctheta (angle increment)

1.732   :n[1].rl (real part of n1)          Prism
0       :n[1].im (imaginary part of n1)
```

```
0.1726  :n[2].rl (real part of n2)              Gold
3.422   :n[2].im (imaginary part of n2)
500     :d[2] (Thickness of phase 2)

1.47145   :n[3].rl (real part of n3)            Layer
0         :n[3].im (imaginary part of n3)
20        :d[3] (Thickness of phase 3)

1.0     :n[4].rl (real part of n4)              Depletion Layer
0       :n[4].im (imaginary part of n4)
30      ;d[4] (Thickness of phase 4)

1.333     :n[5].rl (real part of n5)            Medium
0         :n[5].im (imaginary part of n5)
```

CWater40Dep

```
5       :num_phase (number of phases)
6328    :lambda (wavelength)
0       :begtheta (starting angle)
120     :endtheta (ending theta)
.1      :inctheta (angle increment)

1.732   :n[1].rl (real part of n1)              Prism
0       :n[1].im (imaginary part of n1)

0.1726  :n[2].rl (real part of n2)              Gold
3.422   :n[2].im (imaginary part of n2)
500     :d[2] (Thickness of phase 2)

1.47145   :n[3].rl (real part of n3)            Layer
0         :n[3].im (imaginary part of n3)
20        :d[3] (Thickness of phase 3)

1.0     :n[4].rl (real part of n4)              Depletion Layer
0       :n[4].im (imaginary part of n4)
40      ;d[4] (Thickness of phase 4)

1.333     :n[5].rl (real part of n5)            Medium
0         :n[5].im (imaginary part of n5)
```

CWater50Dep

```
5       :num_phase (number of phases)
6328    :lambda (wavelength)
0       :begtheta (starting angle)
120     :endtheta (ending theta)
.1      :inctheta (angle increment)

1.732   :n[1].rl (real part of n1)              Prism
0       :n[1].im (imaginary part of n1)

0.1726  :n[2].rl (real part of n2)              Gold
3.422   :n[2].im (imaginary part of n2)
500     :d[2] (Thickness of phase 2)

1.47145 :n[3].rl (real part of n3)              Layer
0       :n[3].im (imaginary part of n3)
20      :d[3] (Thickness of phase 3)

1.0     :n[4].rl (real part of n4)              Depletion Layer
0       :n[4].im (imaginary part of n4)
50      ;d[4] (Thickness of phase 4)

1.333   :n[5].rl (real part of n5)              Medium
0       :n[5].im (imaginary part of n5)
```

10.7 Data Analysis

After the trials are complete, open Microsoft Excel and open the collected LabVIEW data. Copy and paste the data from all the different trials into one spreadsheet. Make sure everything is included in the spreadsheet and labeled well. Find the dip angle for the air trial and subtract that value from each angle in the water and methanol trials to get the adjusted angles for both air and methanol. Average the intensity for each angle across all the trials for water and then for methanol. Divide the average intensity at each angle by the highest recorded intensity to scale the data. (The highest intensity at any angle should then be one.) Plot the water trial with adjusted angle on the dependent axis and the adjusted intensity on the independent axis. Do the same for methanol. Use only one graph.

Open the data from the sprangle file. Subtract the dip angle for the theoretical air trial from the angles for the water and methanol data to get the adjusted angles. Graph the theoretical data for the methanol trial, the water trial with no depletion layer, and the water trials with the different depletion layer thicknesses all on the same graph.

Find the difference between the dip angle for experimental methanol trials and theoretical methanol trials. Then determine which theoretical water trial provides the same difference when compared to the experimental water trials in order to find the thickness of the depletion layer.

10.8 Helpful Hints and Advice for Future Students

Mixing the KOH bath with aluminum is never a good idea. Putting glass pipettes in the bath cleans them, but takes the markings off the glass as well.

If APT Config does not recognize both motors, check that the power cords (especially for the red motor) are completely plugged in first. If that does not work, unplug everything, wait a few minutes, plug everything back in (red before orange), and try again.

If there is an issue getting a watertight seal, check the o-ring and make sure it is still in good condition.

Do not take the lenses out of their holders unless absolutely necessary, as they are extremely difficult to get back in correctly.

Read other physics comps. Have people read your comp.

Always bring snacks with you; just don’t eat them in the lab.

Find minions.