

Interfacial Engineering

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Lecture-15

Demonstration of optical tensiometer

Optical tensiometer; ST; contact angle

Welcome back. In today's video lecture, we will look at the demonstration of an optical tensiometer. In the last video lecture, we looked at the force tensiometer. As you know, a force tensiometer is used to measure the interfacial tension of various plates. Okay, using the Wilhelmy plate method or Du Noüy ring method.

Today, we are going to look at the optical tensiometer, which is a complementary technique to the force tensiometer. Sometimes, an optical tensiometer is also called a goniometer. In Greek, gonio means angle, and meter means metric. Collectively, it is called a contact angle meter.

So one can say goniometer also as or call it optical tensiometer also or goniometer also right. So in today's lecture, we will look at the overview of the goniometer followed by the demonstration by one of my PhD students.

OK Let's begin.

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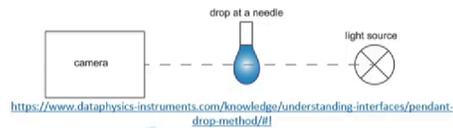
Optical Tensiometer/Goniometer



An optical tensiometer is an instrument used to measure the surface tension of liquids and the interfacial tension between two liquids. Also utilized to measure static, dynamic and roughness corrected contact angles.

The main components of the optical tensiometer:

- A camera.
- A dispenser to dispense a drop.
- A sample stage.
- The light source to illuminate the drop on the sample stage.



The optical tensiometer typically works based on one of several methods:

- **Pendant Drop Method**
- **Sessile Drop Method**
- **Bubble Pressure Method**

Advantages:

- Non-destructive
- Small sample volume
- Easy liquid handling
- Versatility

Limitations:

- Sensitivity to resolution
- Setup Complexity



Interfacial Engineering

As just now described as we just now described so optical tensiometer can also be used right in place of a force tensiometer to determine the surface tension so not only surface tension you can also perform contact angle measurement using the Sessile drop method in addition to the surface tension using the pendant drop method. Sometimes bubble pressure tensiometer is also used to determine the surface tension. So, that is nothing but a bubble pressure tensiometer.

So, essentially it consists of the following components. It has got a camera which fits at the one end and you have the dispenser okay usually a syringe filled with the liquid you know of interest and then so using that one can perform pendant drop as well as Sessile drop method. Then you have got a sample stage so this sample stage can be moved up and down manually or automatically okay then you have got a light source so basically this light source will you know will help you to track the captured droplet image right on the sample stage, okay? So, what are the main advantages of this method and how does it usually function one of the main advantages, I mean, there are several advantages of using an optical tensiometer. You can see that this is often done in non-contact mode, which means that basically this experiment and this device help you determine the surface tension or contact angle using an image. By imaging, I mean using the imaging method you can say that this is also a non-destructive method, and unlike your force tensiometer it doesn't require a huge sample volume so all you need is just a few microliter samples right and easy to handle. You know, unlike the force tensiometer, you know, you won't need to worry about the probe that we use.

Let's say we don't manipulate our Du Noüy ring. Okay. Both are made up of know

platinum or special alloyed material. First, we need to ensure cleaning right after every cycle but in this case, you don't need to worry about those factors there are certain limitations because this method is image-based technique so one would need to worry about the sensitivity to resolution while capturing the image and also setup complexity which means that every time one has to perform the calibration. There is something called baseline correction and there is something called the calibration height with the calibration so this needs to be performed whenever we use this goniometer right so this is one of the these are a few imitations of this technique. So how it works it has got a camera and it has got a light source and you have a needle filled with liquid of your interest so if you want to perform the pendant drop method one has to slowly generate the droplet okay because when you generate droplet two forces are acting on this droplet one is the gravitational force, another one is the surface tension force. Because liquid wants to vent the surface, it will be strongly held by the needle at the tip of the needle. On the other hand, there is also gravity acting downward. So these counteracting forces make the droplet a little bit distorted. So, they will not display a spherical but display like a pendant. The droplet will be in a pendant shape mostly because of these forces acting on the liquid. Because this is not exactly spherical, so one cannot use the constitute relation as such. So, one needs to modify according to the shape factor. So, one has to use the shape factor here. The other one that you can easily perform is the Sessile drop method.

In this, you will have to the droplet has to be placed on the surface of your interest, and then the side view of the droplet will be captured using the camera fitted at the side and then performing the baseline correction and using the curve fitting. There are several curve fitting methods used. Based on curve fitting one can calculate or determine the contact angle. You can see here there is a syringe kept over here. This can be operated manually also or automatically. In some of the latest optical tensiometers, you also have an additional module called pulsating module using which we can generate surface pressure versus area data which will help us calculate the I mean perform the interfacial biology analysis you know, based on interfacial rheology.

So, which means that one can find out what the G' or G'' of the colloidal particles absorbed at the interface. So interfacial rheology can be performed using this module. So, some of the latest techniques available today you know provide these facilities, these modules.

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Demonstration of optical tensiometer

Surface tension measurements can be conducted optically by analyzing the shape of a pendant drop. The configuration of the drop suspended from a needle is governed by the equilibrium of forces, prominently featuring the surface tension of the liquid under investigation.

The surface or interfacial tension can be related to the drop shape.

$$\gamma = \Delta\rho g R_0^2 / \beta$$

Where γ is the surface tension, $\Delta\rho$ is the density difference between fluids, g is the gravitational constant, R_0 is the drop radius of curvature at the apex, and β is the shape factor.

A bubble pressure tensiometer is used for determining the dynamic surface tension. The maximum internal pressure of a gas bubble which is formed in a liquid by means of a capillary is measured.

The internal pressure p of a spherical gas bubble (Laplace pressure) depends on the radius of curvature r and the surface tension σ .

$$\sigma = \frac{(p_{max} - p_0) \cdot r}{2}$$

Source: <https://www.biolinscientific.com/blog/pendant-drop-method-for-surface-tension-measurements>

So next we can look at how this pendant drop method is carried out. So, as I told you shape factor beta is very important if you want to determine the surface tension. So, that is readily determined using the various geometric parameters that are shown in this pendant drop schematic. Once the beta is calculated, software calculates beta based on this pendant shape, based on these geometric factors, you can determine surface tension easily from this relation. In addition to it, we also have what is known as bubble pressure tensiometer. We also looked at a bubble pressure method when we described the various measurement techniques.

This is one of the measurement techniques that we already covered when we discussed contact angle measurement techniques or surface tension measurement techniques. This device helps us perform dynamic surface tension measurements. By calculating the maximum internal pressure of a gas bubble, we can calculate the surface tension using this relation. Alright, so this schematic we already have seen during one of our lectures in module 2. So now we will stop here, but one of my students will explain to you about the optical tensiometer by demonstrating using some liquid samples.

Thank you.

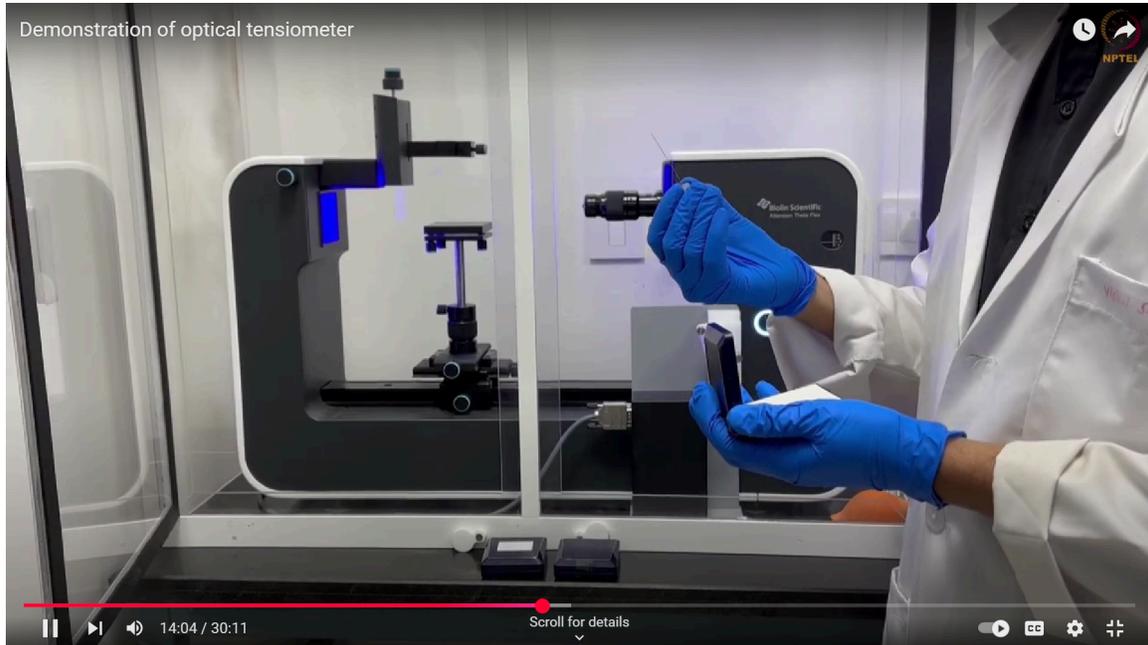
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Welcome back today we will see the working of the goniometer or you can see the optical tensiometer this is our optical tensiometer made by scientific attention. There are mainly three components or we can say the basic components as you can see here one is the camera attached to it or in parallel there is a light source attached to this. Another component is the stage where you have to put your sample and the hanging cantilever where you will hang your syringe. To adjust the tilting of the camera, we have an adjusting facility. By rolling this ruler, you can access the tilt angle of the camera, especially for sessile drop measurement, when you are going to measure the contact angle using the sessile drop measurement. So in that case we have to keep this camera at minus two degree you can actually see the surface and the formed droplet on the surface with the help of that you can calculate contact angle of the system also this stage can move manually like this you can move it up and down it manually. You can adjust the z-axis y-axis and x-axis one by one using these tools which are readily available in the system. You can also adjust the height of the cantilever beam for hanging the syringe now let us move forward to see the software interface for the system as you can see here this is the software named One Attention, where after clicking you will open it is the main software window you can choose test accordingly whether you want to do a sessile drop, batch sessile drop, pendant drop, meniscus dynamic contact angle, pulsating drop here I am going to show this sessile drop for the measurement of the contact angle of a surface using water. After clicking the sessile drop application, you will see this type of interface. In this system, you will get different types of needles that can be attached to this range. Each and every needle has a different edge and diameter that you can choose according to

your sample. Suppose you have highly viscous or less viscous, you can choose accordingly.

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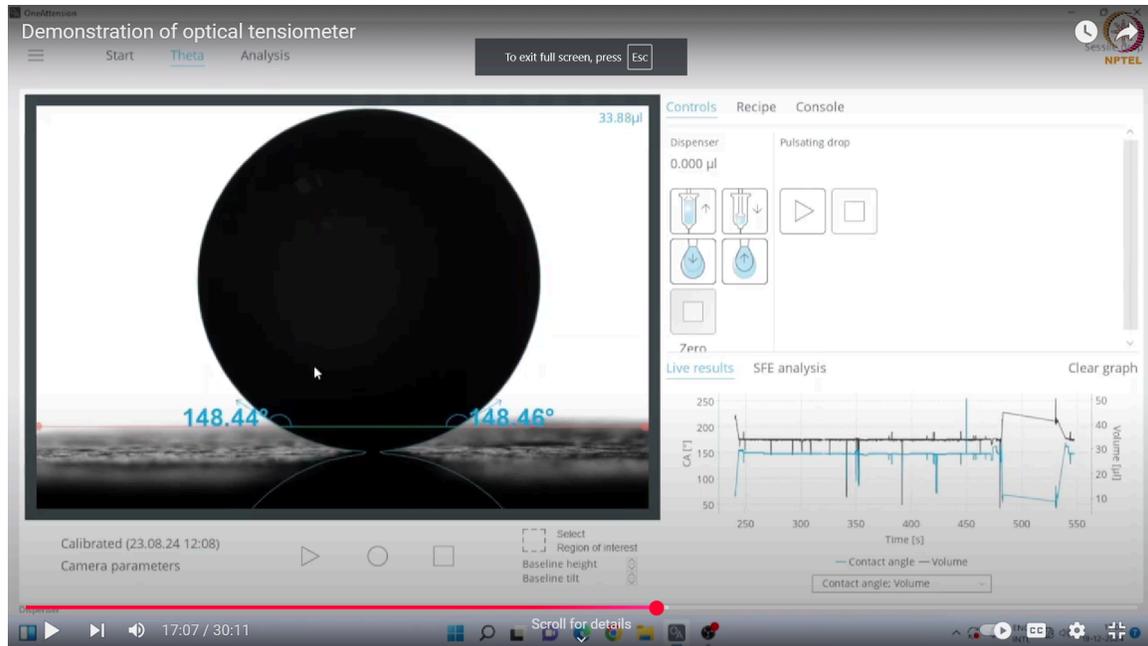
This depends on the applications. You can see here, this is a smaller one with a lower burst needle. The minimum size is like this as you can see here we have another one that is a little bigger than this, this is the bigger one with a bigger diameter needle apart from this if someone wants to measure the interfacial tension of an oil in water system as you know the density of oil is less than water if you put the drop tight inside water it will float upward towards the interface in this case this J type needle or you can say U-hook shaped needle will be used to measure the interfacial tension.

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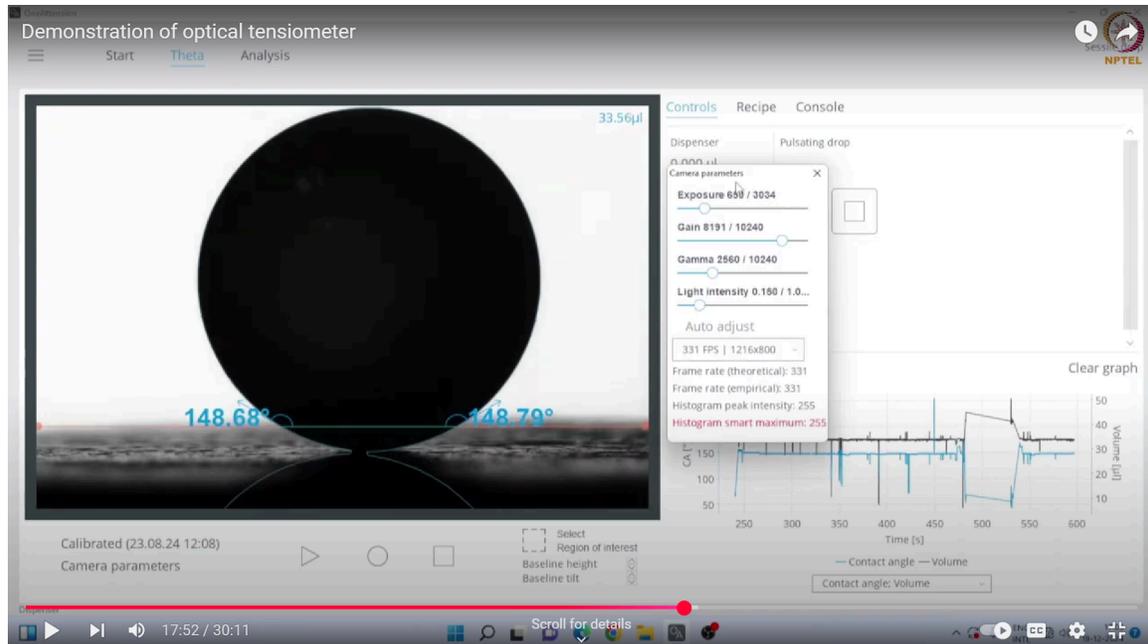
All these needles can be set in different syringes available. Here I am showing you the manual operating syringe. This is made by Hamilton. This also can be used with the help of a syringe pump. To use this syringe, just rotate it manually. By rotating, you can open it and close it. You can also see it here. Or we have one more syringe needle. To use this syringe, just rotate it manually. By rotating, you can open and close it. You can see here, this moves by rotating it anticlockwise or clockwise. This will drop liquid. Now, let us see how we measure the contact angle of our glass. Right here, I will show you the contact angle measurement of a glass slide to understand whether the particular glass has a hydrophobic or hydrophilic nature. I am using a normal glass slide available for microscope purposes with the help of water to determine the hydrophobicity or hydrophilicity of the glass slide. Remember, before doing any experiment, whether it is for contact angle, interfacial tension, or surface tension, we must calibrate the system. For calibration, I am using an iron ball. Just keep this ball in the middle of the stage here. You can see here, that I am keeping this. Now, you have to see, with the help of the camera, remember for calibration, as I told you for drop measurements, we have to maintain the tilt of our camera at -2. Now we will rotate at -2 by rotating the roller clockwise you can change the tilt angle of your camera I am setting this at -2. As you can see here this is the scale that shows the tilt of your camera so here the scale is at -2 now our concern is set at a -2-degree tilt angle. Now let us look at this software to see how this ideal setup looks and how to calculate our system as you can see here our stage is not visible now by changing the z-axis of the stage.

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I am trying to adjust the ball within the frame you can change the minor focus in your camera to position this wall in the middle of the window. Now by adjusting the minor focus, you can properly focus on the ball once the focus is adjusted the entire wall will be visible. After focusing on the ball at this stage you can see that our ball is now properly visible in the system remember calibration is complete when the contact angle formed by the ball is showing 148 points something with respect to its radius on the surface your system will be calibrated you can also change the camera parameters from here.

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By clicking the parameter button, a window will pop up. You can adjust the exposure, gain, gamma, and light intensity. You can also command the system to auto-adjust the camera parameter. So, I am clicking on the auto-adjust option. Now you can see that it is auto-adjusting the light exposure, gamma and everything else. Now our system is completely focused and the camera parameters are fully set. Now click on the calibration. Now remember we are using a ball with a diameter of 4 mm which is a standard one provided by the system you can also use needle calibration if you are using different conditions now by clicking the calibration you can see that the system is showing that is calibrated. Now let us move toward the contact angle measurement using the sessile drop measurement

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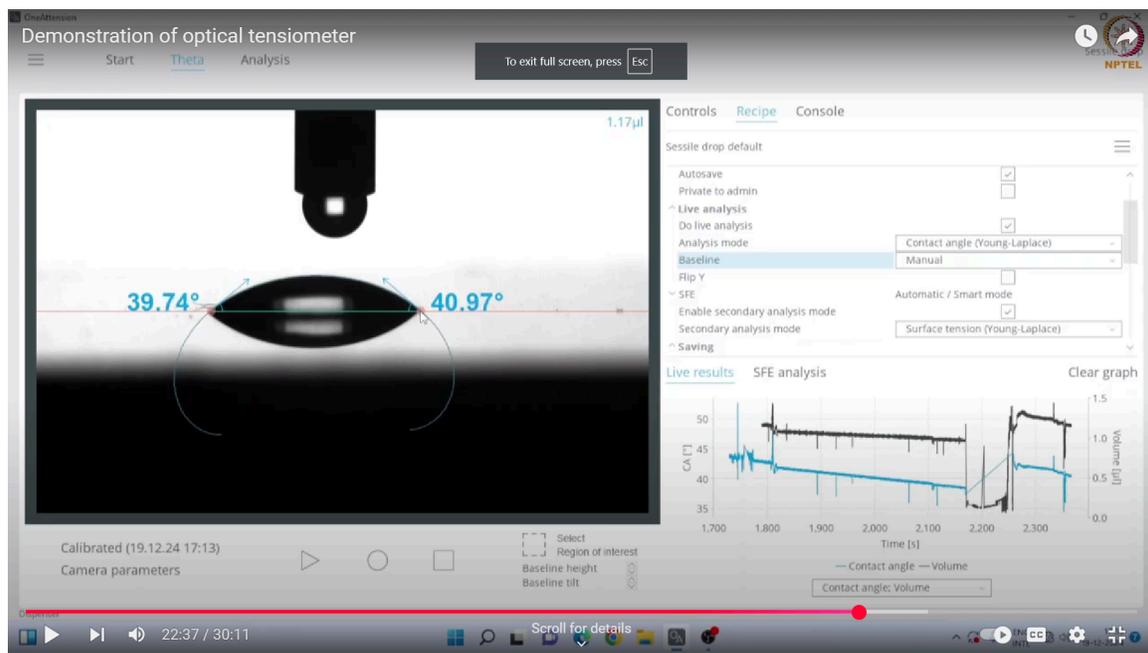
Now you can see where this is the manual syringe, I am going to use I am going to use this is the gauze needle for these manual settings. Now let us see how to refill our syringe by dipping the needle into the water I am rotating the screw to fill the water into the syringe as you can see here, we have sufficient water right now put the sessile drop measurement now you can hang your syringe like this on the cantilever beam

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I'm going to use a glass slide as you can see here this is the standard microscopic glass slide you can use your sample as well remember for sessile drop measurements especially try to use the flattest surface possible so that you can measure the contact angle accurately. I'll place the glass slide on this stage now our glass slide is standing on the stage by adjusting the zx position of the stage I am aligning the height of the glass side so that it is easily visible in the software window you can also adjust the minor focus here by using the screw focusing screw on the camera you can reset and fine-tune the focus as needed using the adjustment. Let us see how glass slides look under the software window. Now we can see the surface of our glass slide.

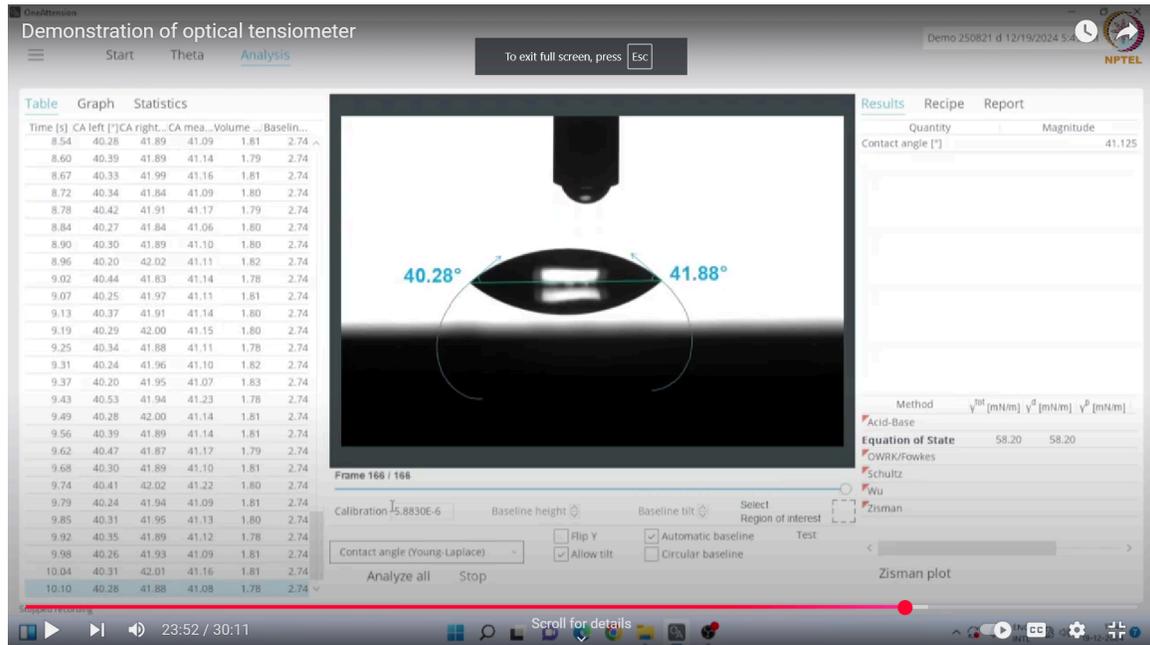
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This is how it looks in the software window when viewed through the camera. Currently, the distance between our substrate or stage and the needle is very high. You can adjust the Z-axis using this control. As you can see here, this is moving downward and this is moving upward. You can also adjust the X and Y direction of the syringe using this screw. So that the droplet will be positioned in the middle of the camera view. Once aligned, let's proceed to sample the droplet. Now let's put the droplet on the substrate. By rotating the screw slowly, you can release the droplet. As you can see here, the droplet is forming and has already been placed on the substrate. The droplet can now be observed in the software window. This is how the droplet looks in the software window. Before starting the analysis, we need to input a few settings. Let me show you the recipe first we will change the experiment name which is the demo one we will put if you want to auto-save your data enable auto-save options you can make the data private as well as enable live analysis if you want to see the results in real-time. You can also choose the

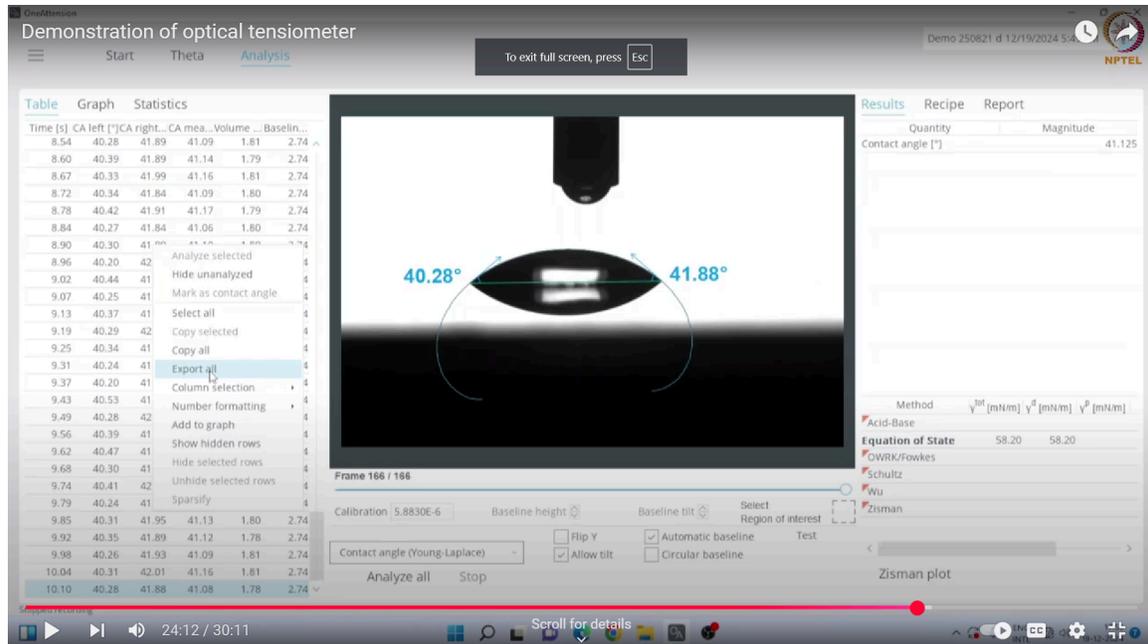
method for contact angle measurement such as contact angle circle polynomial young Laplace for this demonstration I am using the Laplace method . Now let's define the baseline the blue line in the window represents the baseline you can also choose to set the baseline automatically manually or circularly automatic.

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I will be setting the baseline manually adjust the baseline according to the surface like you can adjust it the baseline represents the flat surface of your substrate. If the surface is uneven or has a plateau in certain areas you can manually adjust the baseline by clicking the orange spot and dragging them as needed to reset the baseline, double click the orange spots and the baseline will be straightened. Next, select the phase material that is a light phase and heavy phase here, the heavy phase is water right after conferring the setting perform a minor focus adjustment to make the droplet appear accurately on the surface. You can also fine-tune the camera parameter manually or manually or use the auto adjust feature to optimize settings like exposure you can see here the changes and gamma right now everything is set. Click on the start to begin the measurement the live analysis has started once the contact angle is calculated you can stop the measurement. The software will display the measured contact angle on the substrate by clicking on the analyze you can calculate the average contact angle now in the statistical panel you can see the results including the mean contact angle this one standard deviation that is 0.06 minimum value and the maximum value the results is also visible on the left panel of the screen now to save the data click on the data go to the

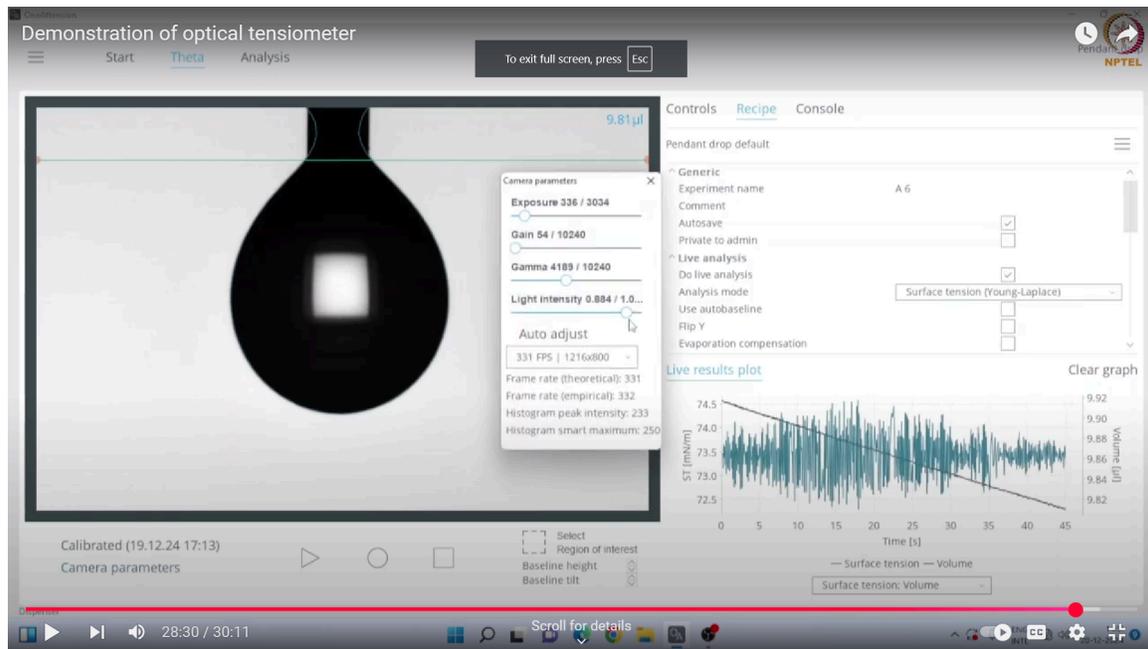
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Export all a pop-up window will appear choose the desired format, and specify the file name I am writing here demo to any desktop your data is now saved and ready for the funder analysis. To save the image of the raw plate for documentation right click on the image and choose the save option so you can see the pictures in two different modes or you can also copy it in the native mode when there is no contact angle shown on the docket, in the overlay mode the contact angle will be visible. So I am saving it in a native mode first nope okay now it is asking in which format bmp jpg png or other format will also come up. I am giving it in the name image123 and saving it somewhere on the desktop .Right now if you want to measure or keep it with the contact angle notations just click on whatever point you want or you can analyze all again. So that it will give you the average contact angle now I can save it and should be the overlay to see this image you can check this is our image with overlay and this is without overlay so this image can be used for the further application purposes. Now let's move forward to the pendant drop measurement as i told you from application window just click on the pendant drop you can see this is how our system and stage looks in the digital position now. Remember first you have to focus your needle properly so that whatever droplet you form is visible and you can also check or remove your stage position so that it does not show any type of attraction with the stage again in this first we have to make a recipe first you have to give a experiment name that is 'm giving T1 and the same auto save option .If you want to make it private you can do it lab analysis is also available choose the one you want to use for analysis mode I am using here the Young-Laplace. Next you can use auto baseline so it will show the auto baseline, if you remove it will show the manually. This you can make the baseline properly . Remember this line will represent the flat edges of your needle also if you want to use slip by condition it will change automatically you can use

evaporation compensation so that if some evaporation is taking place it can prevent it from being too large. You can change the image position as well if you want to record for a longer time you can change it from here I am just recording it for 10 seconds, if you have a syringe pump with this you can change the setting for the syringe pump as well change parameters like the light phase or the heavy phase. Now let's move forward to measure the pendant drop our recipe is ready I already named as T1 like I showed you initially in the sessile drop in the same manner you have to make a drop.

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This is how your drop looks like in the camera after adjusting the focus on its edges a little more for clarity. We will do the auto adjustment here now our droplet is ready as you can see here this is giving us a result of 72.8. This is the blue line for the contact angle, now click on start to visit to see the interfacial tension and this is now recording the interfacial tension of the system. Go to analysis and choose your experiment from the analyzer all you can analyze is your measured interfacial tension now by going here you can see the interfacial tension. Now we will go to the statistical part here you can get your approximate 72.8 which is in good agreement with the available value. Reportedly, people found 72.8 as the interfacial tension for the air-water interface. This is how the base standard deviation is calculated. This is how we measured the interfacial tension of the air-water interface using the Pendant drop technique.

Thank you for joining us.