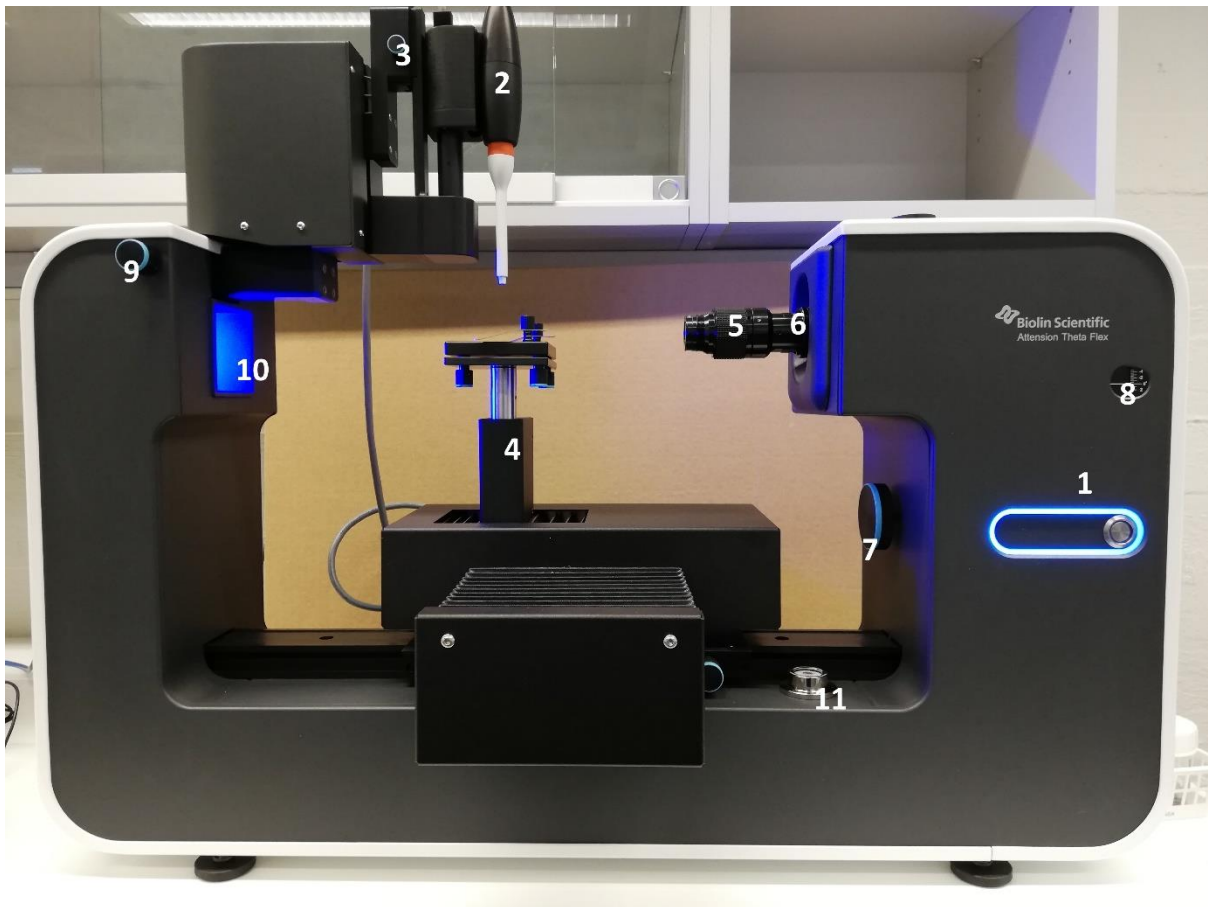


Important parts of the system



- 1) Power button and status indicator light
- 2) Automatic dispenser using disposable tips
- 3) Dual dispenser unit
- 4) Automatic XYZ-stage
- 5) Camera lens focus adjustment
- 6) Camera lens zoom adjustment (**if changed, do a new calibration with calibration ball!**)
- 7) Camera tilt adjustment
- 8) Camera tilt level indicator
- 9) Dispenser lateral adjustment
- 10) Bubble level

1 Introduction

Login

Login to the computer with KKulma (no password). When starting Attension software it will ask for username. Login with your username (Firstname L) and password. Your private measurements will be in the directory DATA/yourusername. Those a set when you attend training, you can later change your password.

About Theta Flex

Attension Theta Flex Optical Tensiometer is a computer controlled and user programmable video based instrument designed for the measurement of:

- Surface and interfacial tension
- Static and dynamic contact angles
- Surface free energy of solids
- Dilatational interfacial rheology

Theta includes a video camera, an automatic XYZ sample stage, dual dispenser unit and a LED light source.

The OneAttension software included with this instrument allows the easy operation of a variety of pre-programmed experiments which can be modified to any needs. The data is stored to the hard drive and can be retrieved and analyzed later. The data analyses can be done without instrument connection. Data files can be easily exported and imported if another data reduction software than the one provided is preferred.

The instrument front panel contains the power button, status indicator (#1) and camera tilt indicator (#8). The instrument status is indicated as follows:

- Indicator light is on when the instrument is ready to be used
- Indicator light spins during recording
- Indicator light blinks when the software asks for input from user

Electrical connections are available on the instrument rear panel. A power cord and USB3 cable need to be connected in order to operate the instrument. The

remaining device ports are used for additional modules. **User should never change any of those connections!**

The rear panel also contains camera linear adjustment lock screw. **Note!** Grip from the optics when adjusting camera linear position. Do not grip from the lock screw. **User should never need to change that!**

The light source (#10) is based on monochromatic LEDs in order to assure a sharp image even for moving objects, with minimal sample heating.

The instrument uses a USB3 camera. The camera has the following image modes:

- 138 FPS @ 1984 x 1264 resolution
- 331 FPS @ 1216 x 800 resolution
- 1002 FPS @ 672 x 373 resolution
- 2003 FPS @ 672 x 136 resolution
- 3009 FPS @ 672 x 57 resolution

The presented frame rates are maximum values. Some functions such as using the live analysis may slow down the actual imaging speed.

The optics has a working distance of 142 – 187 mm depending on focus. Magnification of the image seen on the screen during measurement is changed with the camera lens zoom adjustment (#5). It is advisable to always have as magnified an image as possible to have as accurate results as possible.

NOTE: if you change the zoom level, you have to do a new calibration with calibration ball!

You can also adjust the angle of the camera optics from the knob (#7).

2 Calibration

Turn Theta Flex on and open the OneAttension program. Wait for the computer to recognize the camera before you turn on OneAttension. Notice that when you open the measurement for the first time, Theta will initialize all

the automated functions (e.g. dispenser holder and XYZ-stage). Care should be taken that there is enough space for all the movements to be completed.

Choose measurement method by clicking a measurement icon (sessile drop, batch sessile drop, pendant drop, meniscus, pulsating drop or automatic DCA). Click Adjust Camera Parameters and choose automated camera adjustment by clicking "Auto adjust". You are able to adjust the camera settings also manually if needed.

Place a clean calibration ball and its magnetic holder on the sample stage. Bring the ball into the image on-screen and zoom it appropriately by altering the lens zoom adjustment (#6) and the lens focus adjustment (#5) between the camera and the ball. Adjust the focus ring so that it is midway between its two extreme positions. Next, the zoom must be adjusted. A recommended default zoom setting is such that the calibration ball fills most of the image. Adjust the camera linear position along the base rail until the image is roughly in focus. Lock the position of the camera by tightening the lock screw at the rear panel. Fine-focusing of the image is now possible with the focus adjustment ring. You should find that there is plenty of scope to adjust the focus in both directions. You can use the focus measurement tool by right clicking at the edge of the calibration ball and observing how sharply the light intensity appears to change at the edge of the calibration ball. Press Calibration invalid and accept the calibration ball diameters (standard 4 mm) to complete the calibration. After the calibration is done, do not change the zoom or the calibration has to be redone. Since calibration is fairly easy and simple, it is advisable to do it before every set of measurements.

3 Sessile drop contact angle experiments

Preliminaries

For sessile drop experiments it is recommended to adjust the camera angle to -2 degrees.

When an automatic dispenser (#2) is used, the tubings should be filled. This can be done using the device control buttons. Click **dispense** to expel liquid from the tubing. The dispensing should be done until there are no air bubbles left in the tubing.

When a disposable pipette dispenser is used, the OneAttention software will automatically recognize the dispenser. Please select which tip size is used from the recipe.

Prepare the solid sample and place it on the sample stage. From **Start tab**, choose Sessile drop experiment.

Image Recording

Lift or lower the sample stage (#4) until the solid is visible on the bottom part of the screen. Set the baseline to the sample surface. Lower or raise the syringe with disposable tip (#2) until it is just visible at the top of the screen. Fill in the relevant data on recipe tab. The OneAttention software has default recipes for each of the measurement types. These recipes are suitable for most applications. It might be necessary to add users, liquids or solids to the database manager found in the **main menu**.

Systems with automatic dispenser and dispenser holder: Fill in the experiment name and choose the appropriate drop size, usually 3 – 6 μl . If you want to use 3 μl droplets or smaller, decrease the drop rate to 0.5 – 1.0 $\mu\text{l/s}$. “Volume from image” function is on as a default, and thus the volume of the droplet is calculated based on the image calibration, and not only by using dispenser step motor. “Volume from image” –function is able to provide always the same droplet volume despite possible air bubbles in the needle or the tubings. **Press start**. The measurement is now done automatically.

4 Batch sessile drop experiments

A Batch sessile drop experiment is a set of sessile drop experiments for a batch of samples. Contact angles from a batch of samples are measured successively using the same measurement recipe, and the results can be further analyzed and compared together. This can be convenient if you have lot of similar samples.

Preliminaries

For sessile drop experiments it is recommended to adjust the camera angle to - 2 degrees.

When a disposable pipette dispenser is used, the OneAttention software will automatically recognize the dispenser. Please select which tip size is used from the recipe.

Prepare the solid samples and place the first sample on the sample stage. From **Start tab**, choose Batch sessile drop experiment.

Image Recording

Lift or lower the sample stage (#4) until the solid is visible on the bottom part of the screen. Set the baseline to the sample surface. Lower or raise the syringe (#2) until it is just visible at the top of the screen. Fill in the relevant data on recipe tab. If you want to measure several points per sample define the points in the recipe, or if you have an automatic sample stage set the sequencer accordingly. In batch sessile drop measurements, the image analysis is made from the live image at your chosen points of time. Use the Single point mapper to fill in the points of time you want to use for analysis.

To begin the batch measurement, **press start**. The instrument will measure the first sample, and let you know when it is ready to measure the next sample. Follow the given instructions to measure the rest of the samples. The same points of time and place will be used for the analysis of all samples. When all the samples in your batch have been measured, choose **End batch**.

5 Pendant drop experiments (Manual syringe needed)

A Pendant drop experiment is very similar to a Contact angle experiment in procedure. A note on terms: a surface tension measurement involves a liquid drop and a gas whereas an interfacial tension measurement involves two liquids.

For pendant drop experiments it is recommended to adjust the camera angle to 0 degrees.

Clean the syringe, place the liquid into it and attach it to the syringe clamp. With a standard surface tension measurement, lower the sample stage (#4) so that it is out of the way.

With a standard interfacial tension measurement place the less dense liquid in the syringe, the denser liquid in a cuvette and the cuvette on the sample stage

(#4). Change the syringe needle to a hook needle and attach the filled syringe to the syringe clamp.

From the **Start tab** choose the Pendant drop experiment.

Image recording

Fill in the relevant data in the recipe tab. It might be necessary to add users, liquids or solids to the database manager found in main menu.

With a surface tension measurement simply lower an appropriate drop. With an interfacial tension measurement lift the sample stage (#4) until the cuvette is visible, lower the hook needle into it, lower an appropriate drop and select Flip Y. Press Start or Record from the controls and wait for the images to be recorded.

6 Curve fitting and data analysis

After recording, choose the **Analysis tab** and double click on your experiment. If you wish to analyze a single measurement, accept either the default fitting parameters or adjust them from the Drop analysis window. The software will then process all of the chosen images. The red line represents the baseline, and it should connect the widest points of the curve profile and its reflection to each other. Check this visually, and if unsatisfied unselect Automatic baseline and set it manually. In sessile drop contact angle experiments the baseline is set at the solid surface, whereas in pendant drop experiments it is adjusted at the end of the needle.

By right clicking on the results you are able to perform further data analysis, for example by hiding unnecessary data points. Selected data points can be plotted to a graph or statistically analysed on the Graph and Statistics tabs, respectively. You'll also find the option to export data by right clicking on the results. By right clicking on the drop image you are able to save the separate pictures or the video. Result report in .pdf-format can be also created from the Report tab.

7 Surface free energy experiments

Surface free energy is automatically calculated for each experiment, if enabled on the recipe. The setting can be changed on the recipe and results are shown on the lower right-hand corner after each experiment. Typically, contact angles measured with 2-5 different probe liquids are used in surface energy calculations. On analysis side, different experiments can be combined into an SFE group by choosing them on the analysis window, right-clicking and selecting "Calculate SFE". SFE is then automatically calculated in calculated results. The angles can be also chosen manually from the measurement data by right-clicking on the contact angle and choosing "mark as contact angle".

9 Tips

Recording settings

In **recipe** clicking the text arrow at "Image recording settings" opens you further menu options. You can set different frame rates for recording the images. Remember, that having big FPS for long time results in slower analysis times without giving much better information.

Save your own recipe

When you have made changes to the recipe, save it as private. Then you can load it back another time.

NOTE: There is a bug in software, that keeps both dispensers selected in a loaded recipe even if you chose only "Use liquid 1" when saving the recipe. Other setting of a recipe should be ok.

Copying results from this computer

One obvious way to copy your files is by using the USB drive.

You could use Bluetooth to copy files to you mobile phone or laptop. Pair the device with this computer and send the files.