

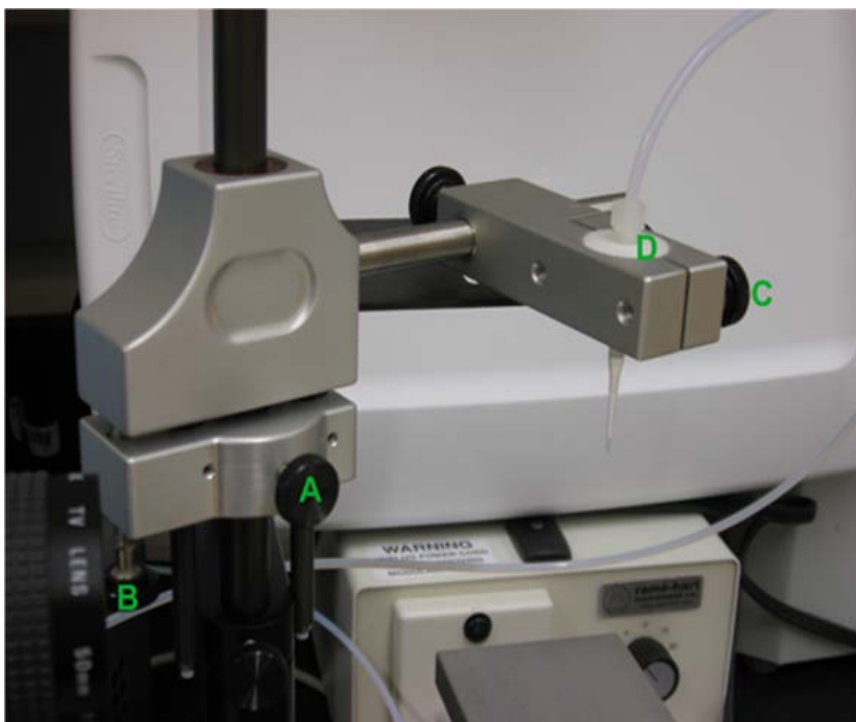
## Materials/Instrument Requirements:

Sample surface should be at least 0.5 x 0.5 cm in size.

Liquids used with the plastic pipette tips need to be compatible with the plastic. If you don't know whether your material will work, contact NBTC staff BEFORE you use the solvent.

The plastic 20 $\mu$ L micropipette tips can be used for contact angle measurements, not hanging drop measurements. The blunt metal syringe tips can be used for either contact angle or hanging drop measurements.

## Instrument parts:



A: Coarse syringe adjustment screw

B: Fine syringe adjustment screw

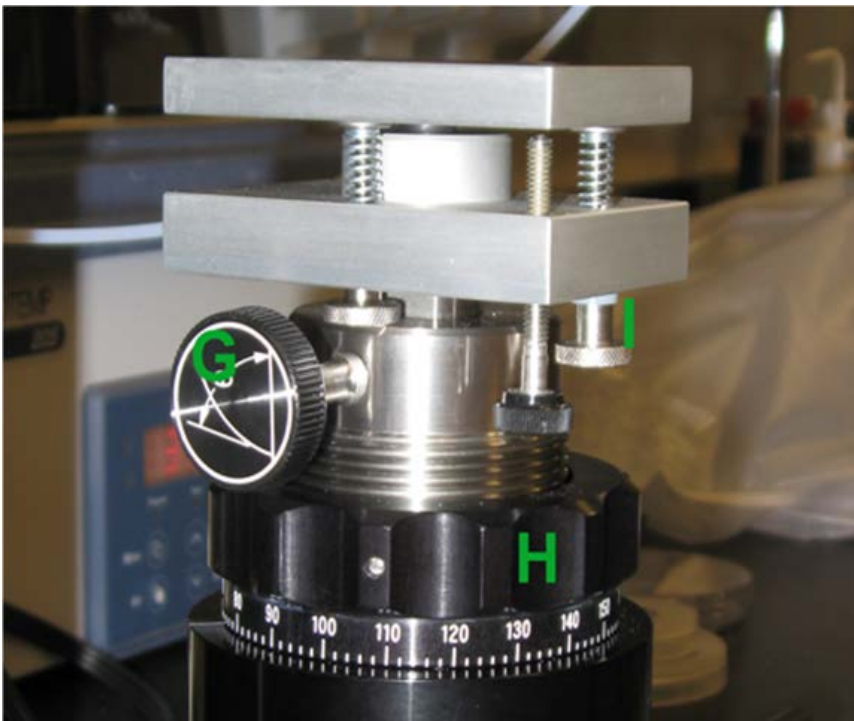
C: Syringe holder screw

D: Syringe holder



E: Focusing knob

F: Left/Right Stage adjustment knob



G: Coarse stage adjustment knob

H: Fine stage adjustment knob

I : Stage tilt adjustment screws

## Initialization:

Log on to the instrument using the central logon computer.

Set up the instrument.

- 1) Remove the dust cover from the goniometer.
- 2) Turn on the liquid dispenser.
- 3) Uncap the cover on the camera and turn on the light to about 50% (So the line is roughly vertical).
- 4) Check that the fine stage adjustment shows about 1cm of threads, and that the fine syringe adjustment screw is about halfway out.

Set up the liquid dispenser.

Place a sample cup underneath the syringe tip to catch any liquid that comes out during dispenser initialization.

Start the Drop Image Advanced program.

“Dispenser not initialized, do you want to Reset now?” YES

-The syringe location will be reset. Some liquid may come out of the syringe.

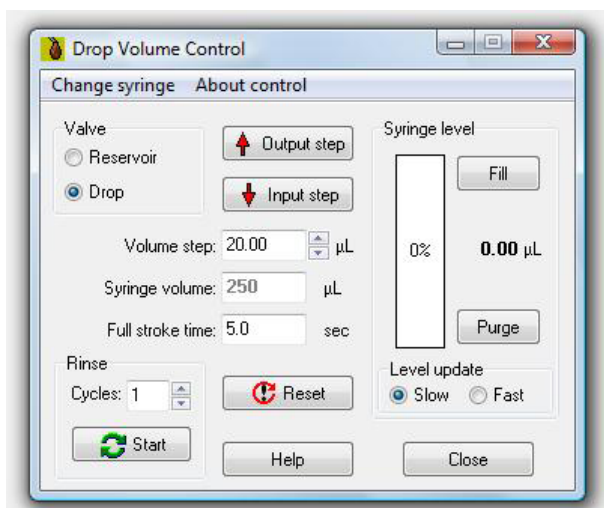
-Upon startup, there should be a Live Image Window visible. If not, go to View→Start Pass-thru so that a live image can be seen.

-If the live image is dark, make sure that A: The light is on, B: the camera is uncapped, and C: The stage is not blocking the light path.

Set up Drop Volume Controller

- 1) Go to Device Control→Drop Volume Control. If Drop Volume Control can be selected, select it and skip to the next step. If not, go to Edit→Options, check “Drop Volume control”, click OK, and Yes when the program asks if it should install and reset the dispenser.

## Drop Volume Control Menu:



**Volume step:** The amount of liquid (or air) that the syringe will draw in when “Input step” is clicked, or which will be expelled when “Output step” is clicked. The volume step can be anywhere between 0.1 and 250 $\mu$ L. Values between 1 and 20mL are typical in many experiments.

**Full stroke time:** Time it takes for the 250 $\mu$ L syringe to travel between both extremes. This will affect the speed of any output or input step, including those programmed in the Event file. The stroke time can be anything from 1-1200s. Values in the 5-30s range are typical in many experiments.

2) Replace the tip on the pipettor, if needed or desired.

3) The syringe can be filled from the open end or from the syringe end. We recommend that you fill from the syringe end using the following method.

- Set “valve” to “drop”.

- Place the syringe into the liquid.

- Click “Fill”.

- Rinse several times by putting a waste cup under the syringe, clicking “purge”, and repeating the steps above.

- Check that the liquid is ready to be expelled by clicking “output step” with the volume step set to about 10 $\mu$ L. A droplet should emerge.

4) Leave the Drop Volume Control window open so that you can add or remove liquid from the surface as needed.

## Taking a Contact Angle Measurement:


Set up the stage:

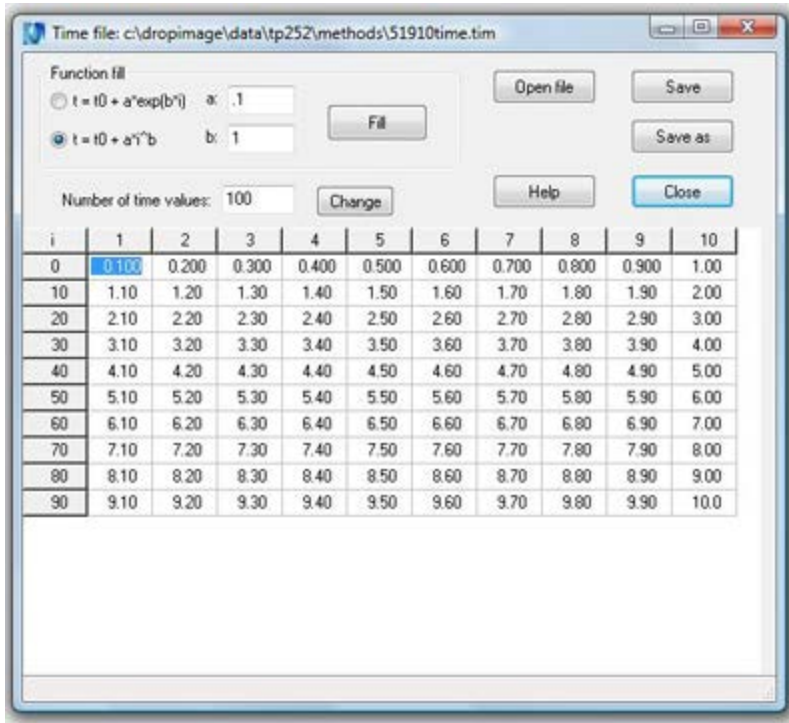
- 1) Loosen the coarse stage adjustment knob while supporting the stage with your hand so it doesn't fall. Lift the stage gently until the bottom of the stage is visible on the live view screen, covering the bottom 5-10% of the live view. The position of the stage can be further adjusted using the fine stage adjustment knob.
- 2) Place your sample on the stage. The sample and stage together should cover less than about 20% of the live image view. Note: If the sample is shiny the edge may not be clearly visible.
- 3) Support the syringe tip assembly with your hand while loosening the coarse syringe adjustment screw, gently lower the assembly until the tip is ~2 mm above the sample, then tighten the screw again.
- 4) Move the stage to the left and right to find the syringe tip. Focus on the syringe tip. The fine adjustment screw can be used to raise and lower the syringe tip until it is fully in view of the screen and focused. For typical measurements, the syringe tip should be centered within the frame, in focus, and quite close to the surface (less than 1mm). If the surface is slanted, the tilt can be adjusted with the tilt adjustment screws.


Choose/Set Up an Experiment:

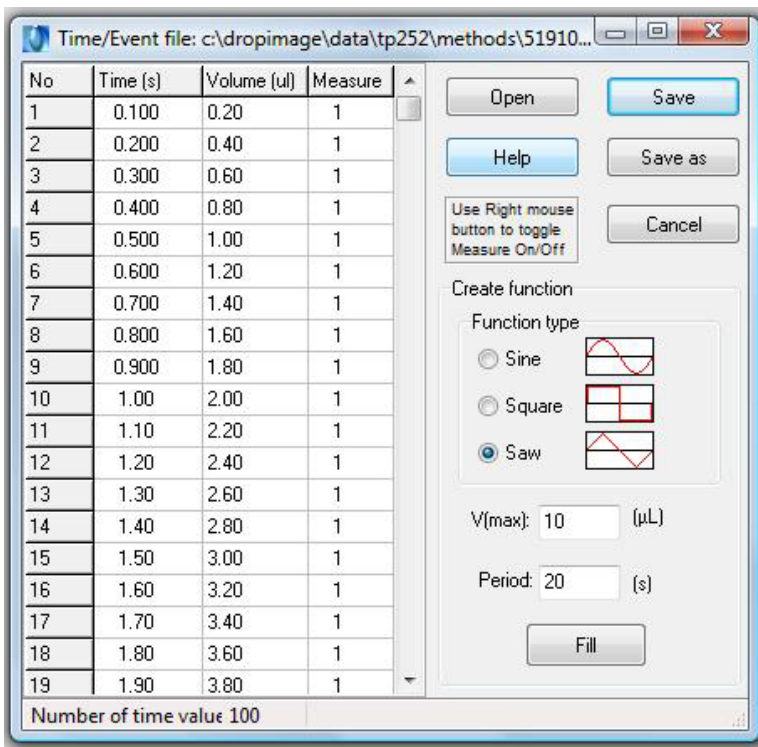
If you have not done so previously, make a personal folder in "User Data" folder on the desktop.

To perform an automated liquid dispensing or take measurements that are not regularly spaced in time, start with step 1. If you do not want to do this, skip to step 3.

- 1) Click on the  ("Time File") and set up the timing and number of data points you want, using the variables shown in the table below. Save As a time file in your folder.



2) Click on the  ("Event File") to make an event file. Load the Time File you want to use. Save As an event file in your folder.



-“Volume” refers to the total amount of liquid expelled or drawn into the syringe since time 0. In the example shown, the liquid will be coming out at a steady rate of 2 $\mu$ L/sec.

-If “measure”=1, it will measure at that time. If you do not want to take a measurement at that time, set “measure” to 0. In the example shown, a measurement will be taken every 0.1 seconds.



3) Create a method by clicking MET on the top menu bar (see image below). When the method is set correctly, Save As a method in your folder.

-Data source: video, unless you are taking data from a previously-run experiment.

-Report: If “screen” is selected, a full report of all data points will pop up at the end of the measurement. This report is in rtf format.

-Data storage: If you want to save every image from which the measurements are taken, choose a file format. If not, choose “none”. Images will be saved with the same file name as you have saved the method—filename[0].jpg, filename[1].jpg, etc.

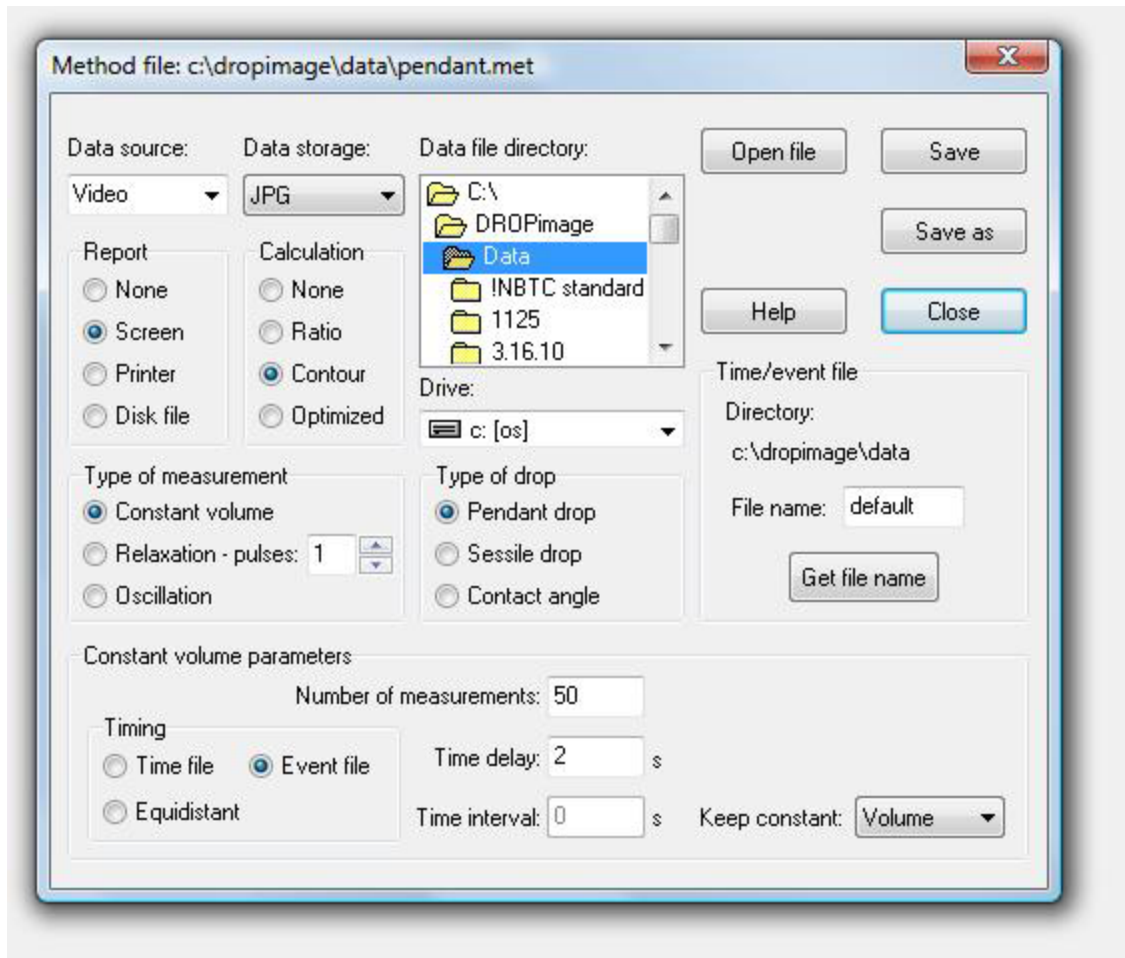
-Calculation: This is the method used to determine the droplet shape accurately. “Contour” is appropriate for most applications. Check the help section for more information.


-Data File Directory: This is where your report and image data will be saved. Use the same folder you have saved the experiments to.

-Type of measurement: “Constant volume” is appropriate for most applications. This instrument does not have an oscillator.

-Type of drop: Choose Contact angle, and click the L and R to measure only the left or right side of the droplet (if the letter is visible, a measurement will be taken).

-Timing: If you saved an event file that you want to use, be sure to click the Timing: Event file button and also load the event file. If you skipped steps 1 and 2, choose Equidistant and fill in the number and frequency of measurements you wish to take.

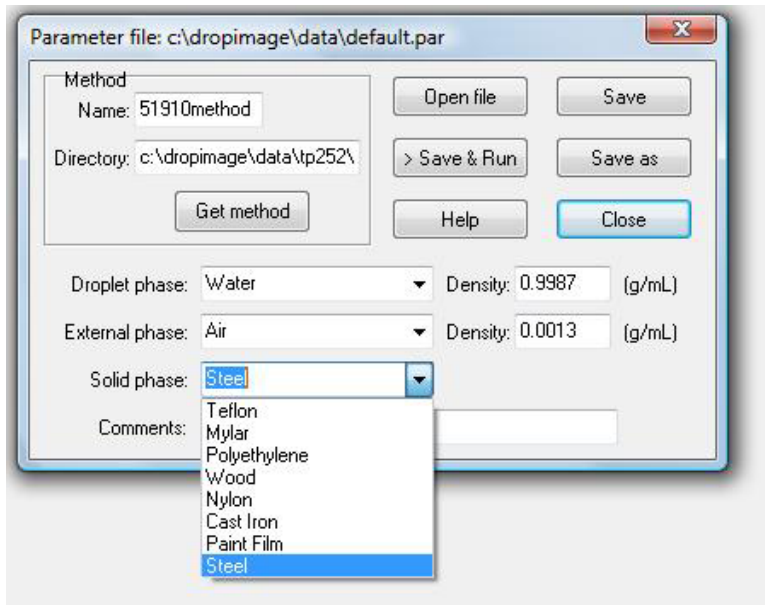


4) Create a parameter file by clicking  PAR on the top menu bar. (See image below.) Load the method you want to use. “Droplet phase” is what’s in the syringe, and “External phase” is the fluid you’re putting that droplet into (typically air). “Solid phase” is the surface. Save As a parameter file in your folder.

-There are two things these settings are used for: 1) The instrument will look at the relative densities of the droplet and external phase to determine whether it is looking for a droplet at the top or bottom of the screen. 2) The materials selected will be displayed on your report or contact angle file if you use either of those. If you are just checking the contact angle, just set the droplet phase to something more dense than the external phase, and don’t worry about the solid phase.

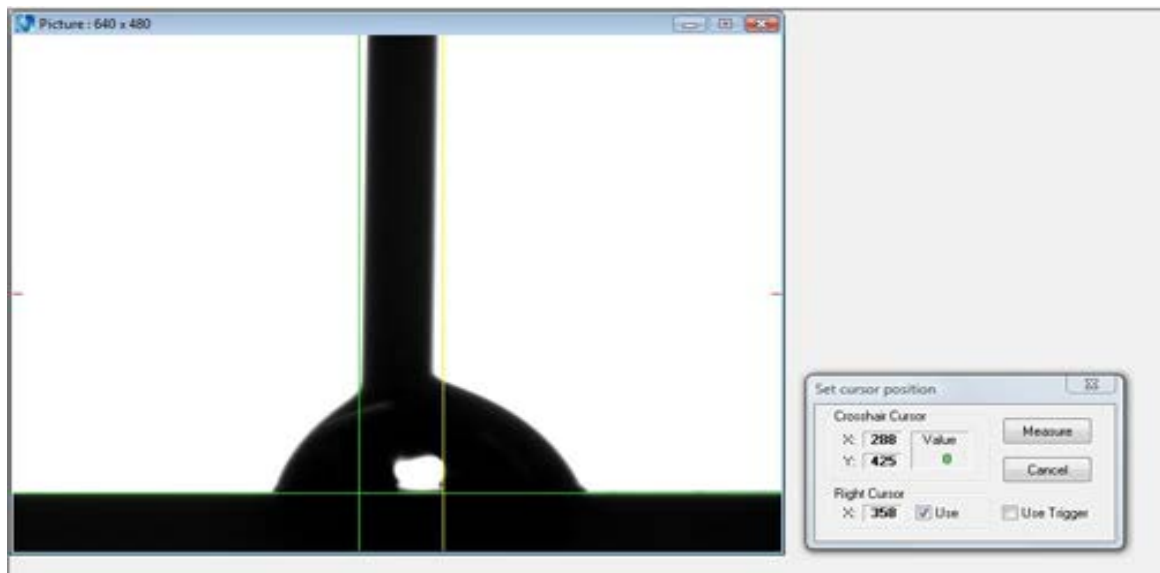
-To get a material added, please contact the tool manager.





Run an experiment:

- 1) Click the  ("Start Active Experiment") to start a measurement.



-The vertical green line defines the left side of the needle and is moved by holding down the left mouse button. The vertical yellow line defines the right side of the needle and is controlled with the right mouse button. The space between these two lines will be ignored in calculating the shape of the droplet.

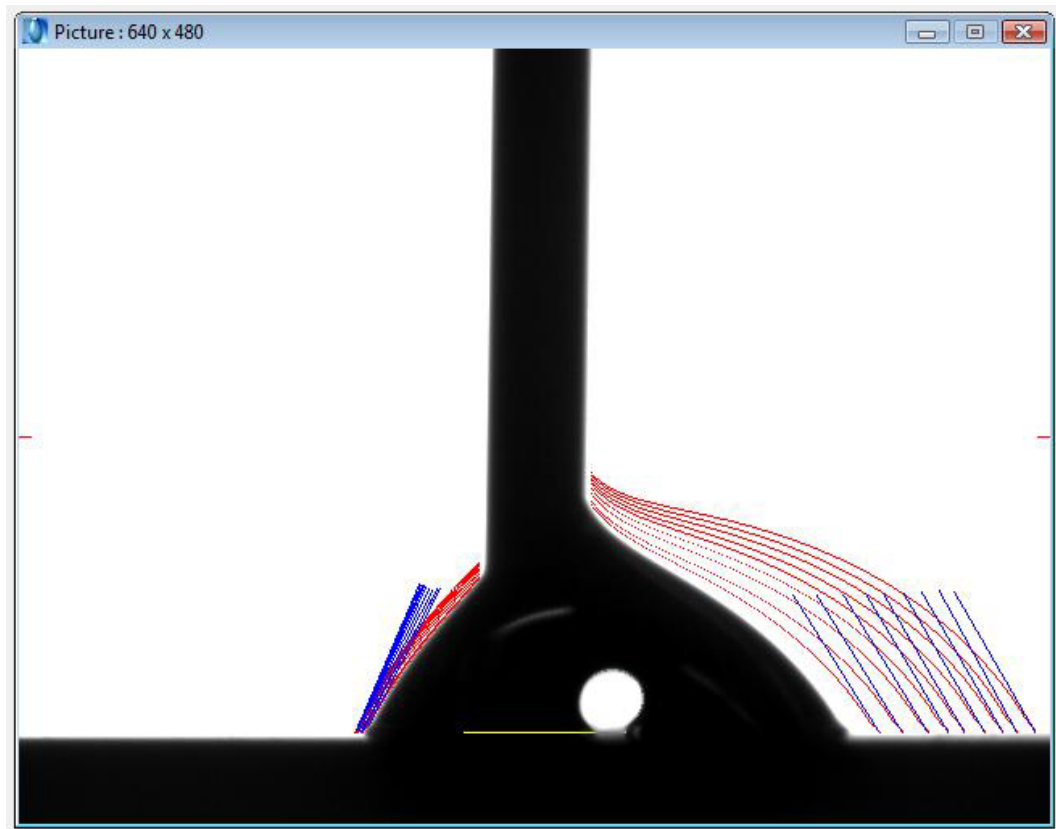
-The horizontal line defines the surface. It is controlled by holding the shift and left mouse button together. Arrange the three lines as in the image above. When ready, click "Measure".

2) A measurement is taken based on your parameter and method files, along with the time and event file if you used them.

- To manually add or remove liquid from the drop use the Drop Volume Controller menu.

-A tracing of the drop and contact angles will show up during the course of the measurement. Verify that the software is correctly identifying the edges of the bubble by looking at the measurement lines. In the example of a receding contact angle measurement shown below, there was an error on one measurement (the yellow line), but the others appear to have been accurate.

-Data is saved in your folder with the filename used in the parameter measurement as "filename.log". Images will be saved as "filename[0].jpg", "filename[1].jpg", etc. A report will be saved as "filename.rtf". These files will be overwritten with the next measurement taken with the parameter file unless saved in a different location, or with a different file name.



## Taking a Pendant Drop Measurement:

Set up the syringe:


- 1) Loosen the coarse stage adjustment knob while supporting the stage with your hand so it doesn't fall. Lower the stage completely, and put a small sample cup or Petri dish on the stage to catch any falling droplets. If you are going to be measuring one liquid in another, fill a cuvette with liquid and lift the stage until the needle is submerged in the liquid.
- 2) Support the syringe tip assembly with your hand while loosening the coarse syringe adjustment screw, gently lower the assembly until the tip is visible on the screen, then tighten the screw again.
- 3) Move the stage to the left and right if needed to center the image of the syringe tip, and focus on the syringe. The fine adjustment screw can be used to raise and lower the syringe tip to a good height. For typical measurements, the syringe tip should be centered within the frame, in focus, and near the top of the frame.

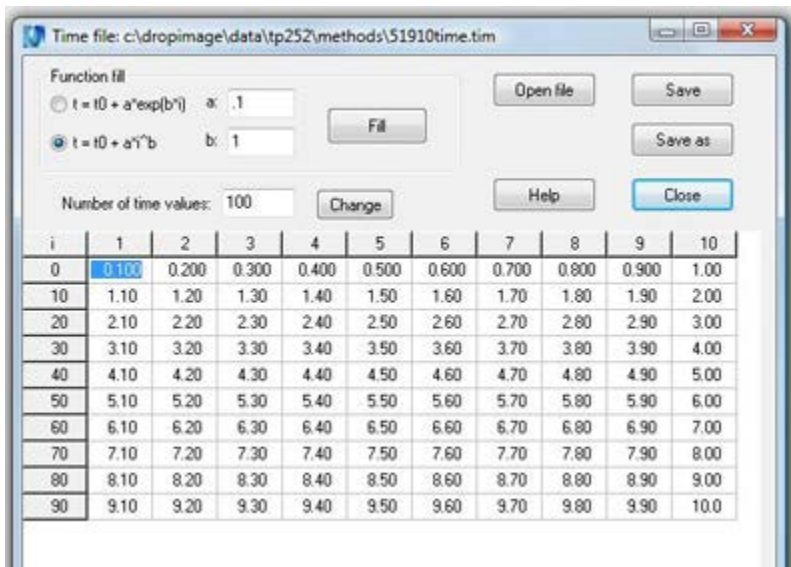
Choose/Set Up an Experiment:

If you have not done so previously, make a personal folder in "User Data" folder on the desktop.

To perform an automated liquid dispensing or take measurements that are not regularly spaced in time, start with step 1. If you do not want to do this, skip to step 3.




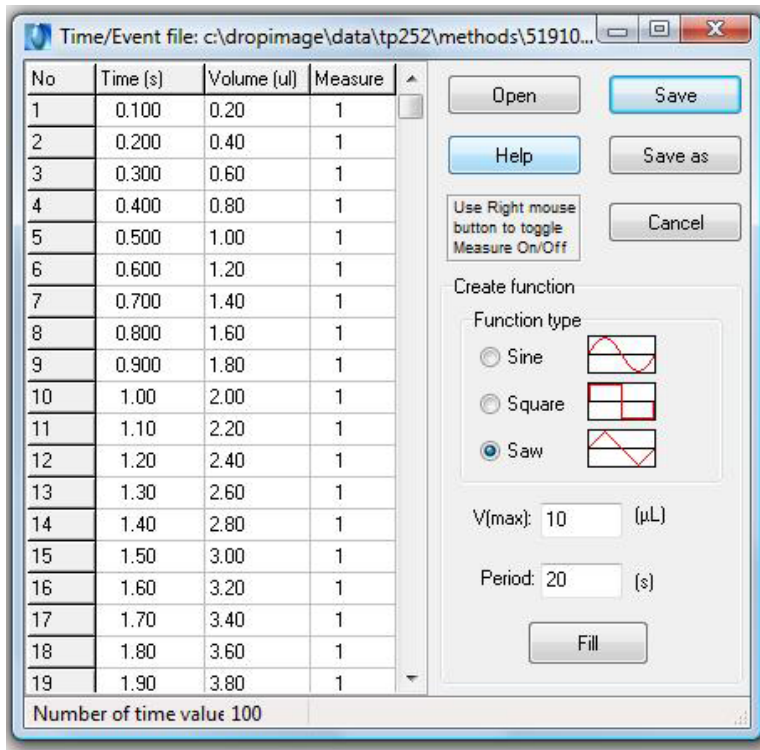
- 1) Click on the  ("Time File") and set up the timing and number of data points you want, using the variables shown in the table below. Save As a time file in your folder.



i	1	2	3	4	5	6	7	8	9	10
0	0.100	0.200	0.300	0.400	0.500	0.600	0.700	0.800	0.900	1.00
10	1.10	1.20	1.30	1.40	1.50	1.60	1.70	1.80	1.90	2.00
20	2.10	2.20	2.30	2.40	2.50	2.60	2.70	2.80	2.90	3.00
30	3.10	3.20	3.30	3.40	3.50	3.60	3.70	3.80	3.90	4.00
40	4.10	4.20	4.30	4.40	4.50	4.60	4.70	4.80	4.90	5.00
50	5.10	5.20	5.30	5.40	5.50	5.60	5.70	5.80	5.90	6.00
60	6.10	6.20	6.30	6.40	6.50	6.60	6.70	6.80	6.90	7.00
70	7.10	7.20	7.30	7.40	7.50	7.60	7.70	7.80	7.90	8.00
80	8.10	8.20	8.30	8.40	8.50	8.60	8.70	8.80	8.90	9.00
90	9.10	9.20	9.30	9.40	9.50	9.60	9.70	9.80	9.90	10.0




2) Click on the  (“Event File”) to make an event file. Load the Time File you want to use. Save As an event file in your folder.



-“Volume” refers to the total amount of liquid expelled or drawn into the syringe since time 0. In the example shown, the liquid will be coming out at a steady rate of 2 $\mu$ L/sec.

-If “measure”=1, it will measure at that time. If you do not want to take a measurement at that time, set “measure” to 0. In the example shown, a measurement will be taken every 0.1 seconds.



3) Create a method by clicking  MET on the top menu bar (see image below). When the method is set correctly, Save As a method in your folder.

-Data source: video, unless you are taking data from a previously-run experiment.

-Report: If “screen” is selected, a full report of all data points will pop up at the end of the measurement. This report is in rtf format.

-Data storage: If you want to save every image from which the measurements are taken, choose a file format. If not, choose “none”. Images will be saved with the same file name as you have saved the method—filename[0].jpg, filename[1].jpg, etc.

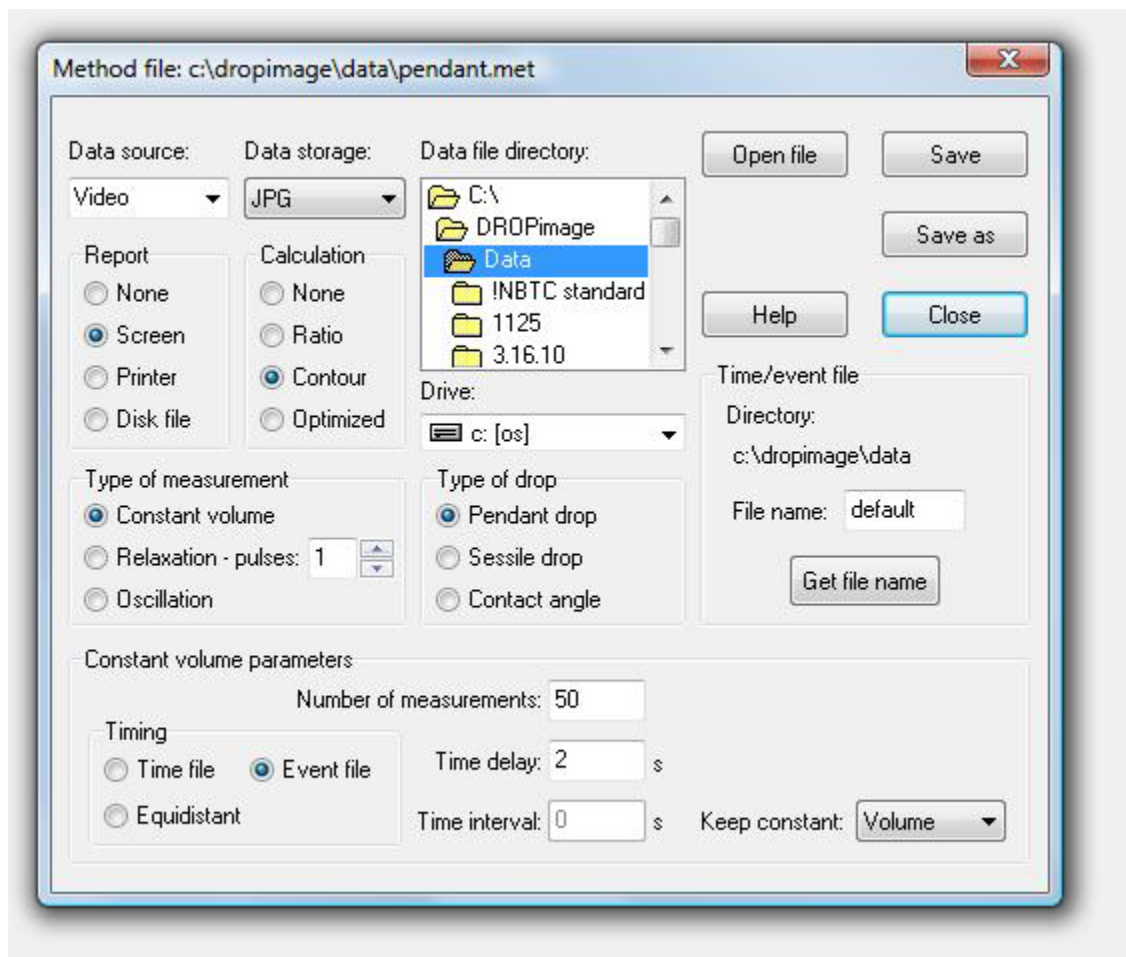
-Calculation: This is the method used to determine the droplet shape accurately. “Contour” is appropriate for most applications. Check the help section for more information.


-Data File Directory: This is where your report and image data will be saved. Use the same folder you have saved the experiments to.

-Type of measurement: “Constant volume” is appropriate for most applications. This instrument does not have an oscillator.

-Type of drop: Choose Pendant Drop.

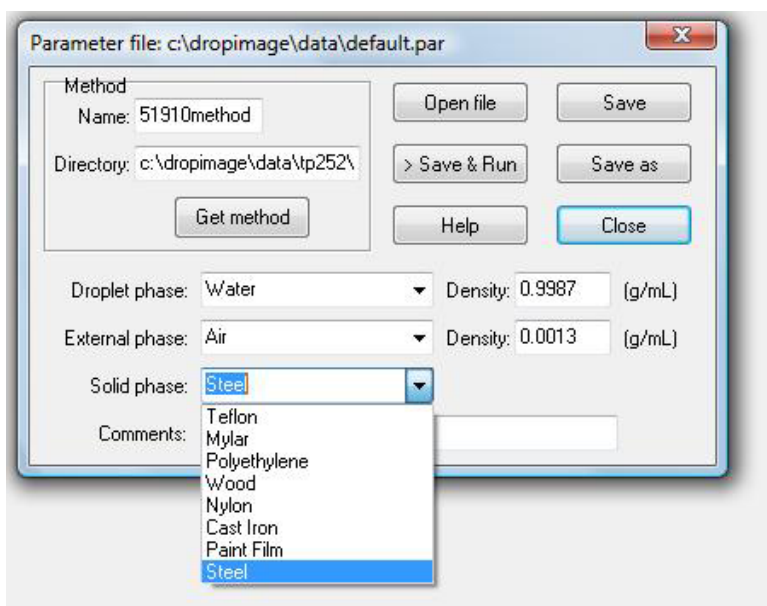
-Timing: If you saved an event file that you want to use, be sure to click the Timing: Event file button and also load the event file. If you skipped steps 1 and 2, choose Equidistant and fill in the number and frequency of measurements you wish to take.



4) Create a parameter file by clicking  PAR on the top menu bar. (See image below.) Load the method you want to use. “Droplet phase” is what’s in the syringe, and “External phase” is the fluid you’re putting that droplet into (typically air). Save As a parameter file in your folder.

-There are two things these settings are used for: 1) The instrument will look at the relative densities of the droplet and external phase to determine whether it is looking for a droplet hanging down from or pushing up from the bottom of a needle. 2) The materials selected will be displayed on your report or surface energy file if you use either of those.

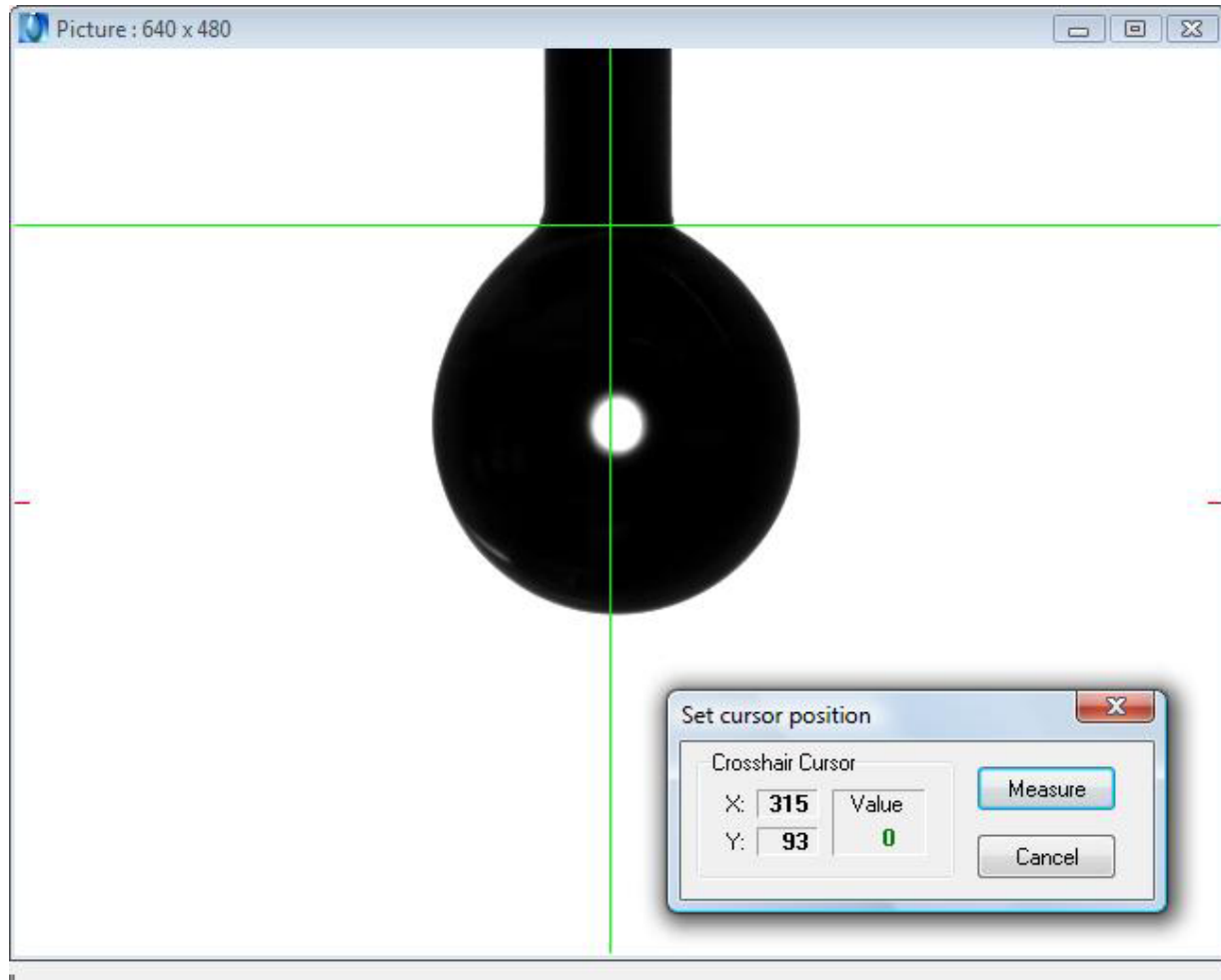
-To get a material added, please contact the tool manager.



Run an experiment:

1) Click the  (“Start Active Experiment”) to start a measurement.

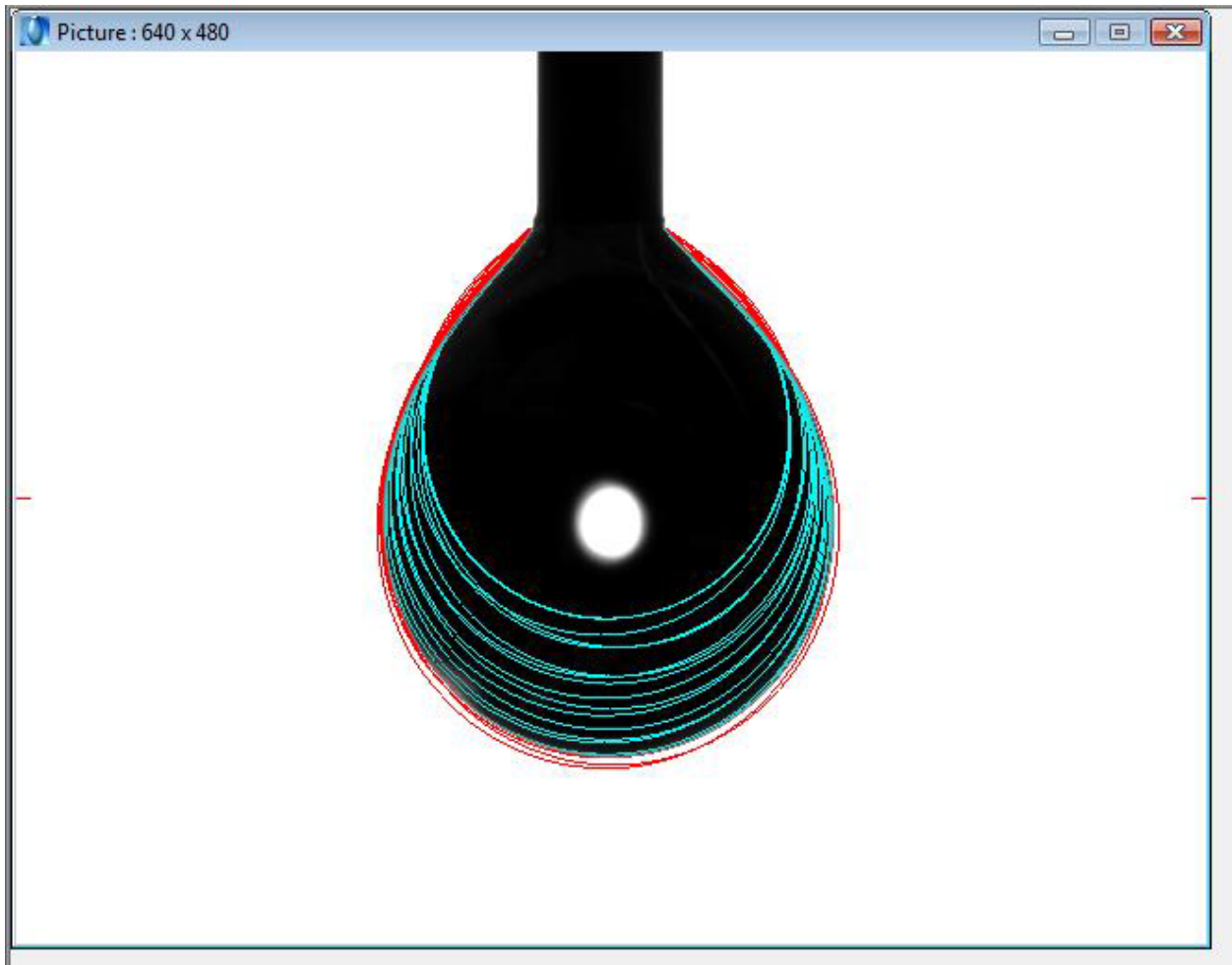
-The crosshairs should be moved so that the vertical line is close to the center line of the needle tip and droplet, and the horizontal line defines the bottom of the needle tip. Its location is moved by holding the left mouse button. Arrange the crosshairs as in the image below. When ready, click “Measure”.



2) A measurement is taken based on your parameter and method files, along with the time and event file if you used them.

- To manually add or remove liquid from the drop use the Drop Volume Controller menu.

-A tracing of the drop will show up for each measurement. Verify that the software is correctly identifying the edges of the bubble by looking at the measurement lines. In the example shown below, the instrument accurately traced the outline of the droplet as it got larger. The lines are not perfectly parallel because room vibration caused the droplet to move slightly during the measurement.



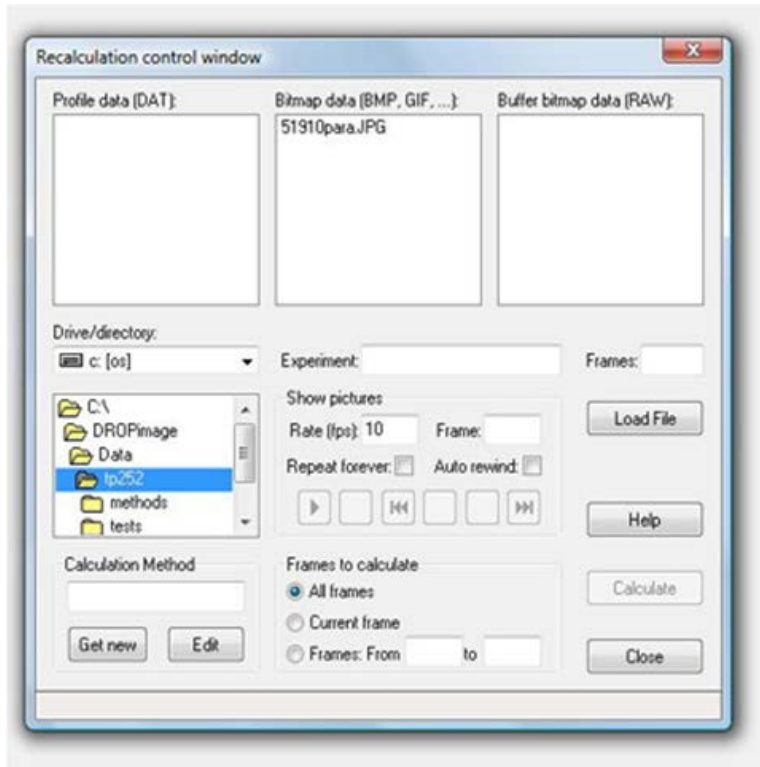
-Data is saved in your folder with the filename used in the parameter measurement as "filename.log". Images will be saved as "filename[0].jpg", "filename[1].jpg", etc. A report will be saved as "filename.rtf". These files will be overwritten with the next measurement taken with the parameter file unless saved in a different location, or with a different file name.



## Other software functions:

### To recalculate a particular data point:

Measurement → Recalculate



Note: unless you save these images in a different folder or with a different name, the next measurement using the same parameter filename will overwrite this data.

### To determine surface energy from a contact angle measurement:

1) File → Make contact angle file

-Open your parameter filename.log, choose a datapoint, and click "add" to add it to the contact angle file. Save As a contact angle file in your folder.

2) CA Tools → your chosen surface energy calculation method.

-File Open—pick the .ca file that you just saved. Pick a solid, then the liquids, and choose your method.

-If you need other materials to be added, please talk to the tool manager.