

TopView Contact Angle Analysis

April 4, 2004

Top view analysis measures the average diameter of the sessile drop when viewed from above, then computes what the contact angle must be in order that the sessile drop volume match the *a priori* known dispense volume.

Diameter Measurement

An edge finder approaches the drop from, in order, the left, bottom, right, and top sides of the image. It specifically finds and ignores needles that enter the image from outside. It shows the points that were found on the periphery by a sequence of connected straight yellow line. It also shows the best circle fit with a dashed yellow line. The straight lines connect the peripheral points in the order that they were found, starting in the upper left-hand corner and working counterclockwise. It is possible for the straight line to “cut across” a large region when no points were found there. It is also possible for it to cross back over itself when the drop periphery is irregular; this is OK and the algorithm does not care in which order the points are found.

The algorithm fits the edge points to a circle using a regression algorithm rather than measuring the major diameter in the X and Y directions. This has the advantage that only a portion of the drop periphery need be seen in order to estimate its radius of curvature and center. Of course, it is better to see the entire drop, but it is not necessary. The diameter of the circle is used to calculate the contact angle.

The Edge Sensitivity control on the Calibration tab will affect whether a marginal point is included or not. You can try different settings and see the consequences in the edge fit that is drawn when you click the Contact Angle command button. Generally speaking, moving the slider to the left will increase the number of points included.

The Top View Asymmetry reported in the Results window is twice the standard deviation of the found points against the regression circle, divided by the regression radius. In general, an Asymmetry less than 1% is very good and over 10% should be considered carefully as the drop is then very irregular.

Magnification Calibration

Magnification calibration is made with a 3mm sapphire ball placed in the field of view (with no glass present). Make a contact angle measurement and use the measured diameter in the Calibration tab under Calibration by Linear Measurement. The actual diameter is 3mm, of course.

Dispense Volume

We will refer to the drop still hanging on the dispense tip as the pendant drop and the drop on the surface as the sessile drop. There are two ways to determine the dispense volume:

- characterize the needle by the procedure described below and enter the average dispensed volume on the Contact Angle tab in the Drop volume text box. With this method, you can take SnapShots of the sessile drop.
- take a movie starting with the pendant drop on the tip and ending with the sessile drop on the surface. The software will calculate the dispense volume from the pendant drop diameter.

Long fused silica needles with the trade name MicroFil are used to dispense liquid in the top view setup. MicroFil dispense needles can be ordered from FTA or World Precision. Smaller needles give smaller drops. However, it is the outside diameter that determines the dispensed volume because the drop clings to the outside of the needle. The best way to determine the average drop volume is to weigh drops using a laboratory balance with 1 milligram resolution or better. Dispense 10 drops into a small boat and then weigh it and divide by 10 to get the average weight. The volume, V, in microliters for the weight W in milligrams of water (with density .9982 g/cc) is

$$V = W / .9982 \approx W$$

On the needles we used for these tests, we obtained

Gauge	OD (um)	ID (um)	Average Volume (ul)
28	350	250	9.80
34	164	100	4.35

The ratio of the volumes is very close to the ratio of the outside diameters (but it is not perfect because the outside diameters are not closely controlled).

- When TopView mode is selected on the Contact Angle tab, any Distance measurement you make on the image is transferred to the Spherical diameter text box. This lets you measure drops just before they detach for their actual volume. If you do not want the drop diameter text box to change, uncheck the TopView mode.

- Whenever the drop diameter text box changes on the Calibration tab, the perceived drop volume box is updated. Make sure you do not inadvertently change the drop volume, particularly when you are using the recommended weighing mode to characterize a needle.
- It is sufficient to characterize needles once and then trust the volume. The secret is to dispense slowly so that the full volume will form before falling off.

An example set of measured pendant drop volumes for a 34GA needle are shown here. The mean is 4.35 μ l and the COV is 2.0%. Note the uncertainty is multiplied by the measured contact angle, not the maximum possible angle. So a 10° measured angle would have a 2% = 0.2° COV from the uncertainty in the dispensed volume.

4.35 μ l	4.27 μ l	4.28 μ l	4.30 μ l
4.28 μ l	4.45 μ l	4.45 μ l	4.45 μ l
4.45 μ l	4.31 μ l		

A factor is required to translate the pendant drop diameter (which is what can be observed and measured) into pendant drop volume. The process is

pendant diameter \rightarrow spherical volume \rightarrow \times factor \rightarrow pendant volume as weighed

The factor for 34GA needles measured at FTA is 1.709. This is entered in the Sphere to pendant vol factor text box on the Contact Angle tab.

If the Contact Angle tab's Auto Measure Dispense Volume box is not checked:

The current volume entered in the Dispense volume text box will be used by the algorithm. There will be no automatic determination of pendant volume.

If the Contact Angle tab's Auto Measure Dispense Volume box is checked:

Starting at the current image in the Movie, the drop diameter will be measured and the pendant volume calculated on subsequent images. When there is a jump in volume, the current average volume is recorded and entered in the Drop volume text box. Subsequent images are regarded as sessile drop contact angle images. If the starting image is the last image in the Movie, as it would be if the Movie were a SnapShot for example, the image is treated contact angle measurement and the existing Drop volume is used.

Measurement Method

If you wish to use a pre-determined pendant drop volume:

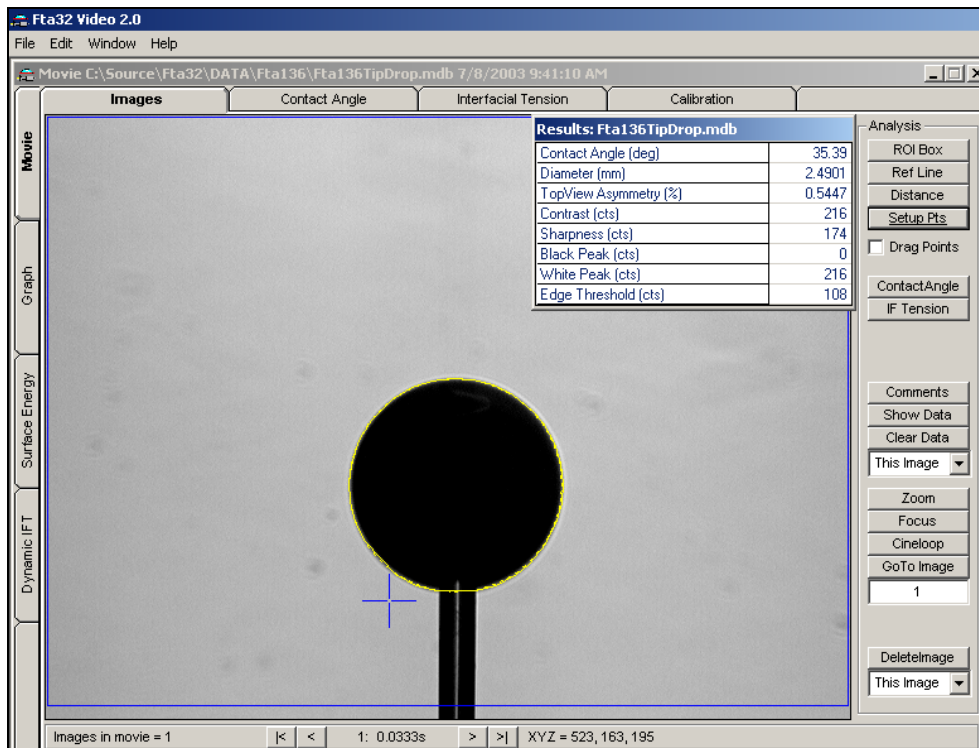
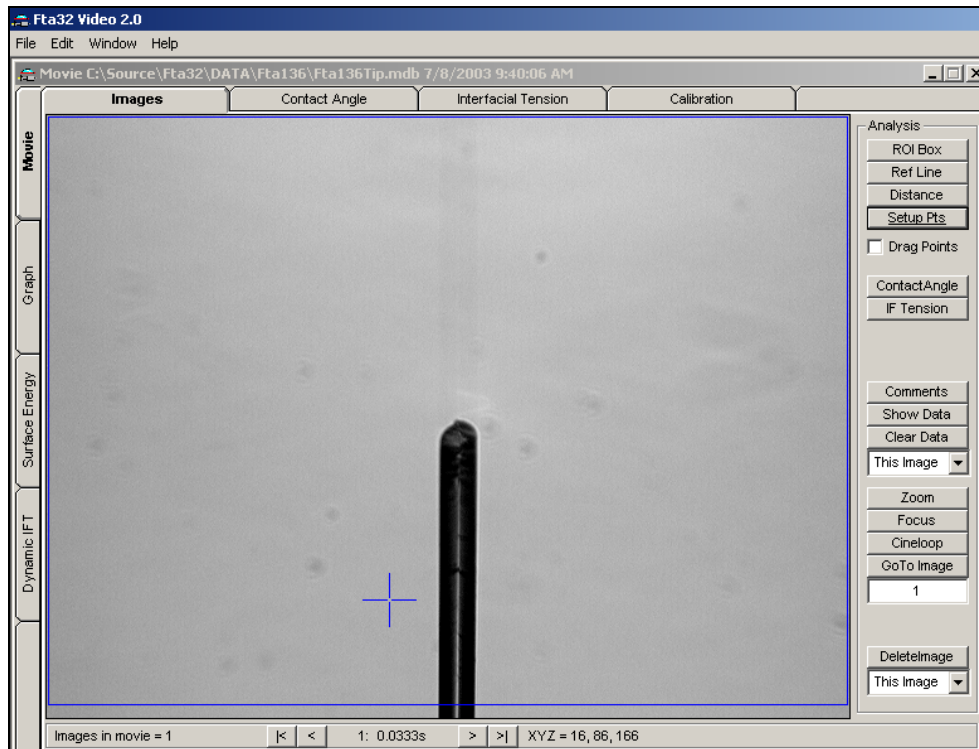
- Uncheck Auto Measure Dispense Volume.
- Ensure the correct volume is entered in the Drop volume text box.
- Use the Video Trigger to trigger a Movie when the drop actually detaches from the tip. Use the checkbox on the Capture tab and right click in the image to place the blue target crosshairs. The Live analysis will Trigger when the crosshairs changes gray scale value. Alternatively, take a SnapShot after the drop detaches.
- The best focus is when the black periphery line is very thin or has a mating white line also. The following figures show actual measured drops and typical images.

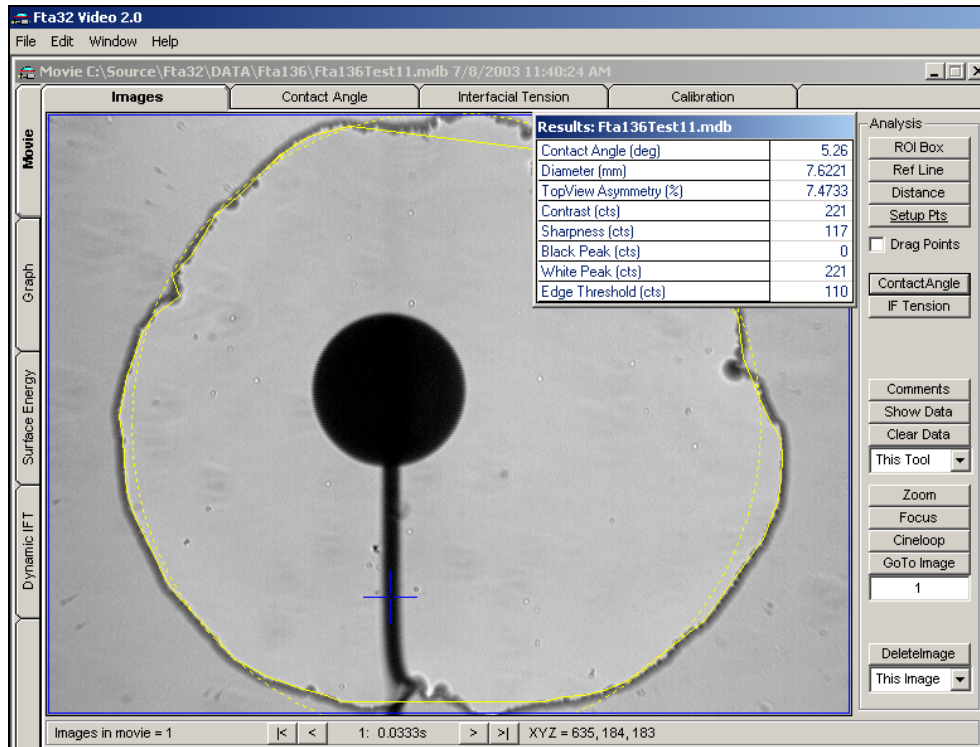
If you wish to measure the pendant volume in real time:

- Check Auto Measure Dispense Volume.
- Ensure the correct factor is entered in the Drop Sphere to pendant vol factor text box.
- Use the Video Trigger to trigger a Movie when the drop actually detaches from the tip. Use the checkbox on the Capture tab and right click in the image to place the blue target crosshairs. The Live analysis will Trigger when the crosshairs changes gray scale value. Furthermore, make sure you have something like 30 images before the trigger at .033s per image. This will give a full second of data prior to detachment.
- A good technique is to turn the Gilmont syringe barrel enough that you are sure a drop will detach, then entirely remove your hands from the instrument. For a 34GA needle that will dispense just a little over 4 μ l, a good amount is 5 μ l. The liquid will take several seconds to flow through the narrow bore needle, and the tip will become very still during that time with your hands away from the instrument.
- The best focus is when the black periphery line is very thin or has a mating white line also. The following figures show actual measured drops and typical images.

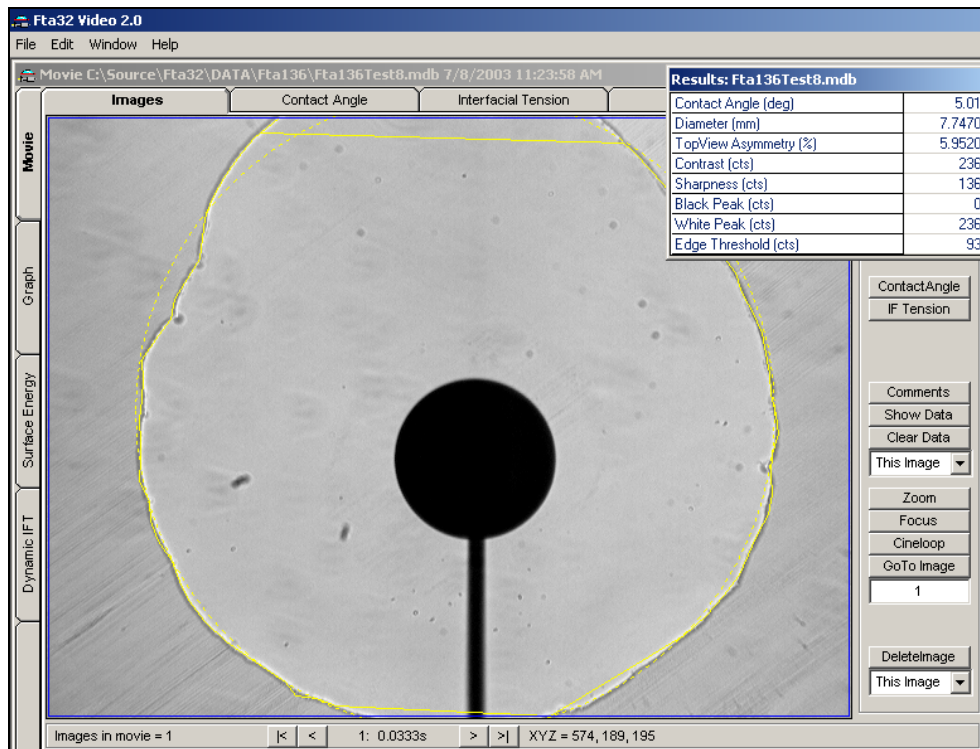
No matter which method you use, always check the images for reasonable fits between the yellow graphics and the drop. If you outliers, spots on the sample, cause bad fits, use the ROI box to exclude them. You can also use Edge Sensitivity to exclude undesirable points and include desired ones.

Example Images

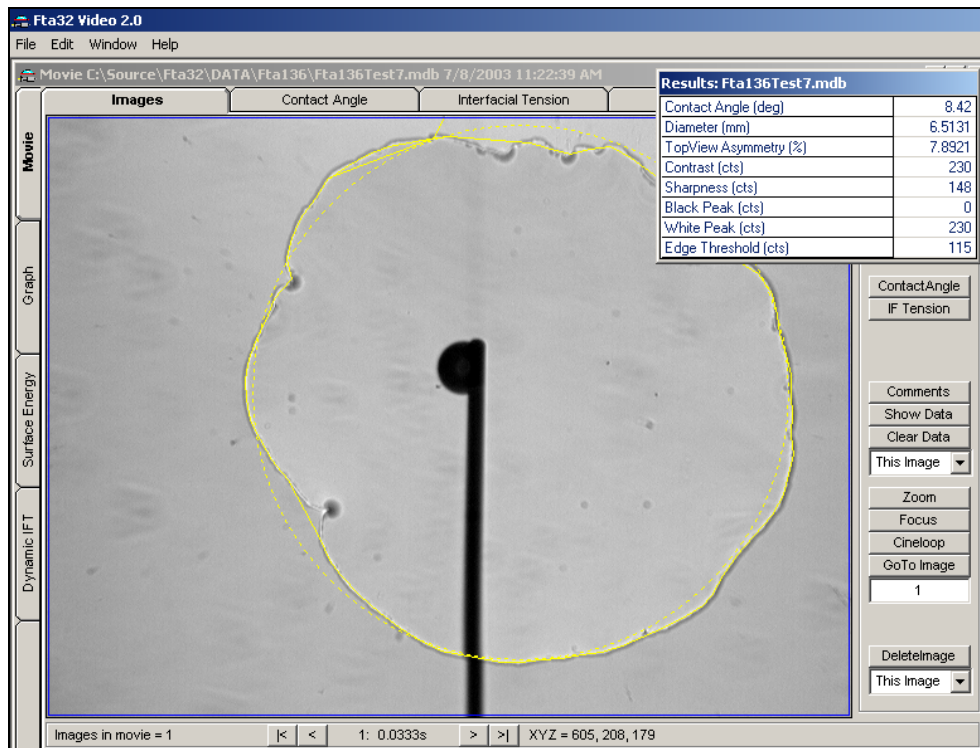




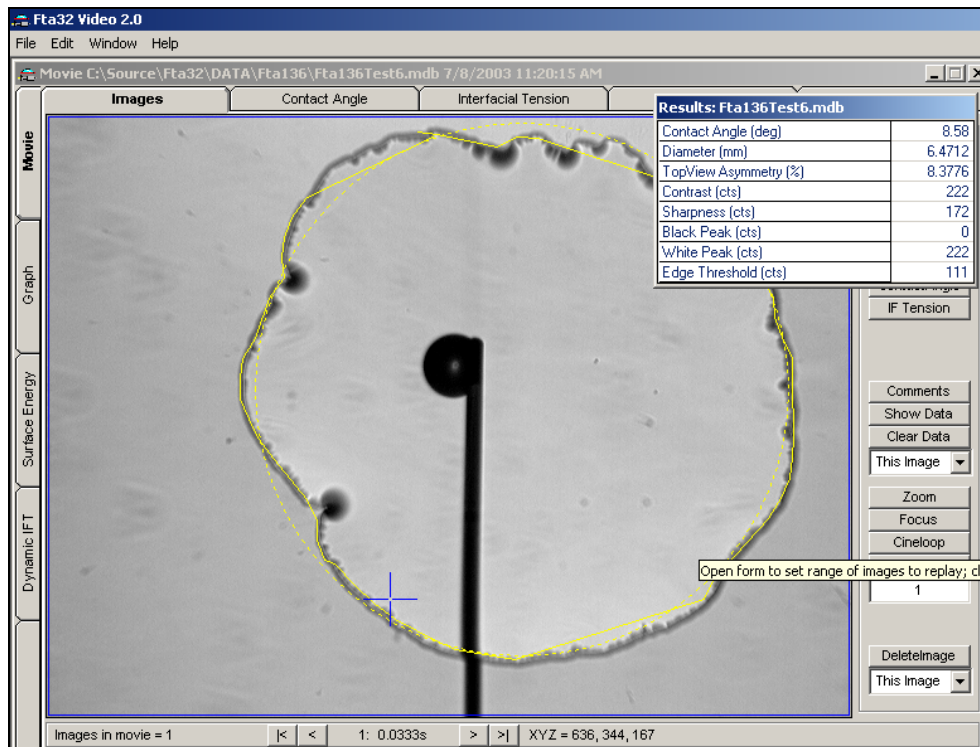
Drop on glass showing black/white boundary at periphery.



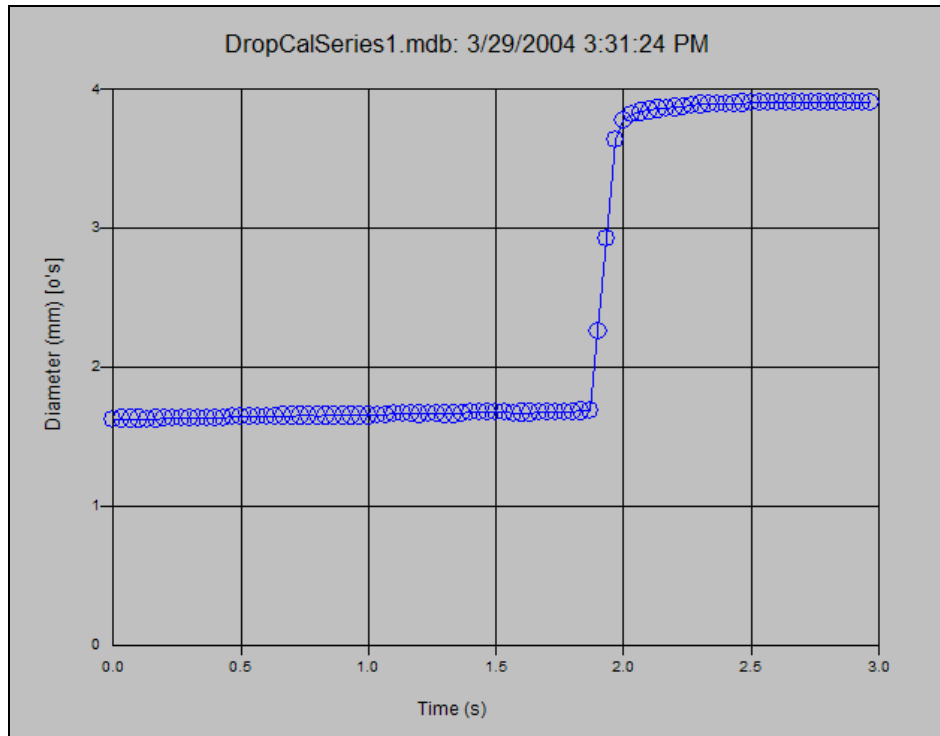
Drop on glass showing black/white boundary at periphery.



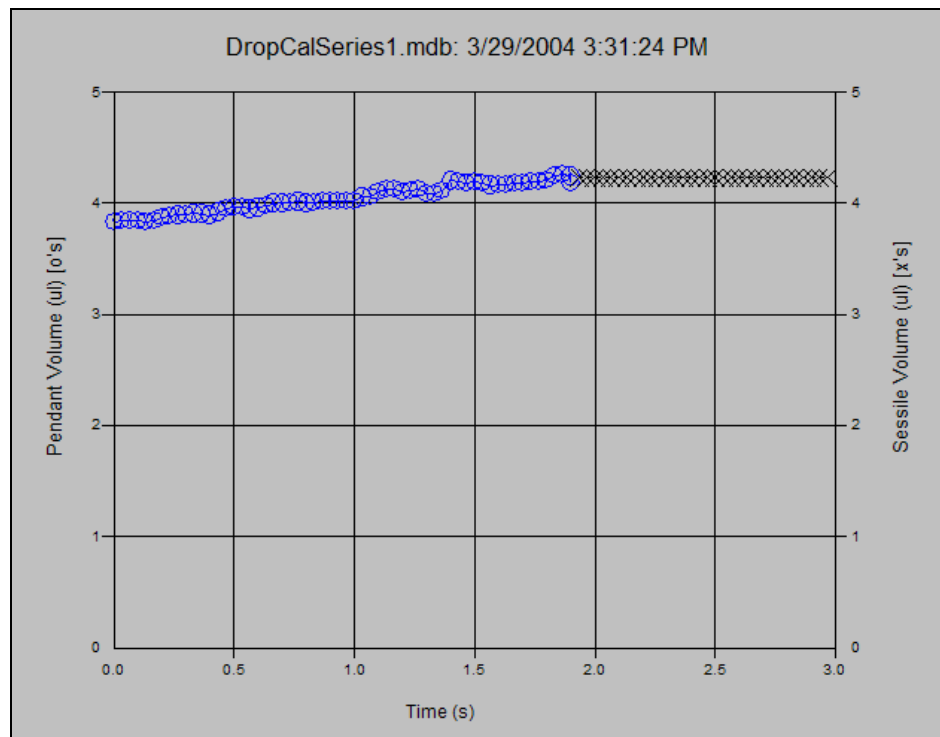
Another example low-angle drop that is well focused



This drop shows more pronounced black periphery.



Typical diameter as a function of time. Jump occurs at detachment.



Calculated pendant (left) and sessile volumes (right).