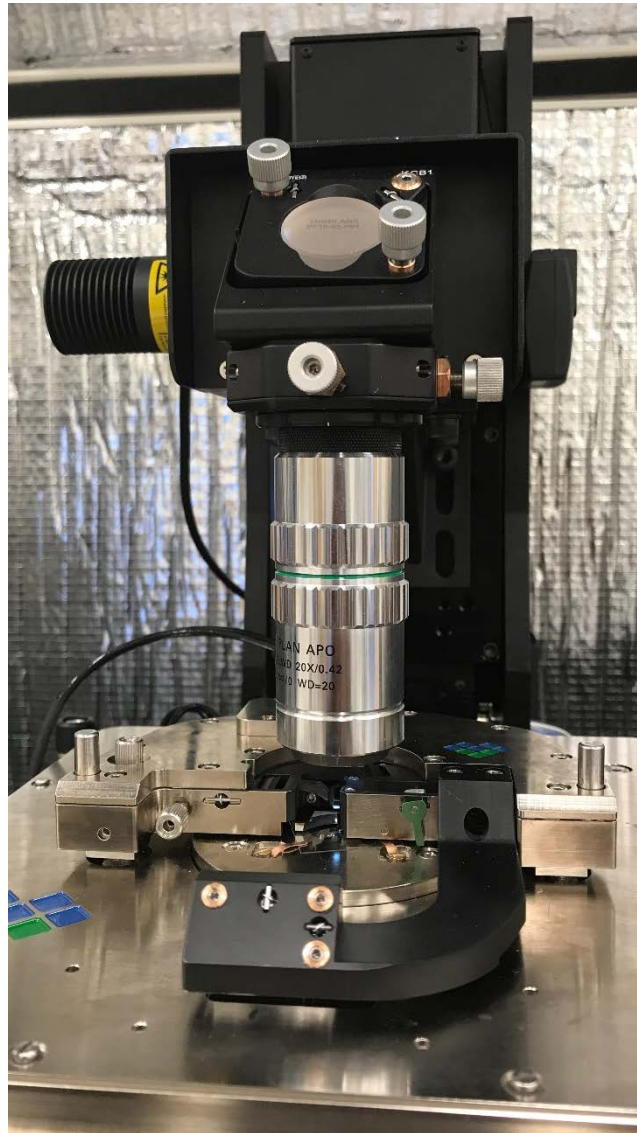


Piffy the Photoinduced Force Microscope

Standard Operating Procedure

June 2017

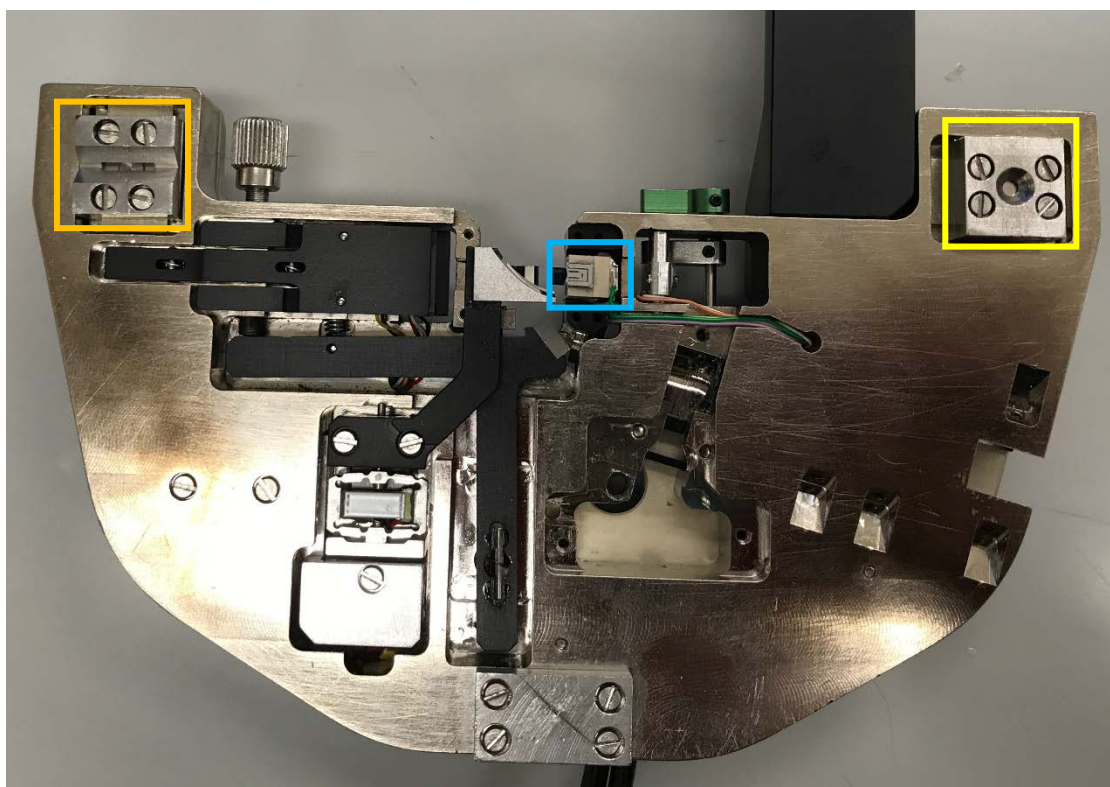
By Clean Energy Fellow Jessica Kong



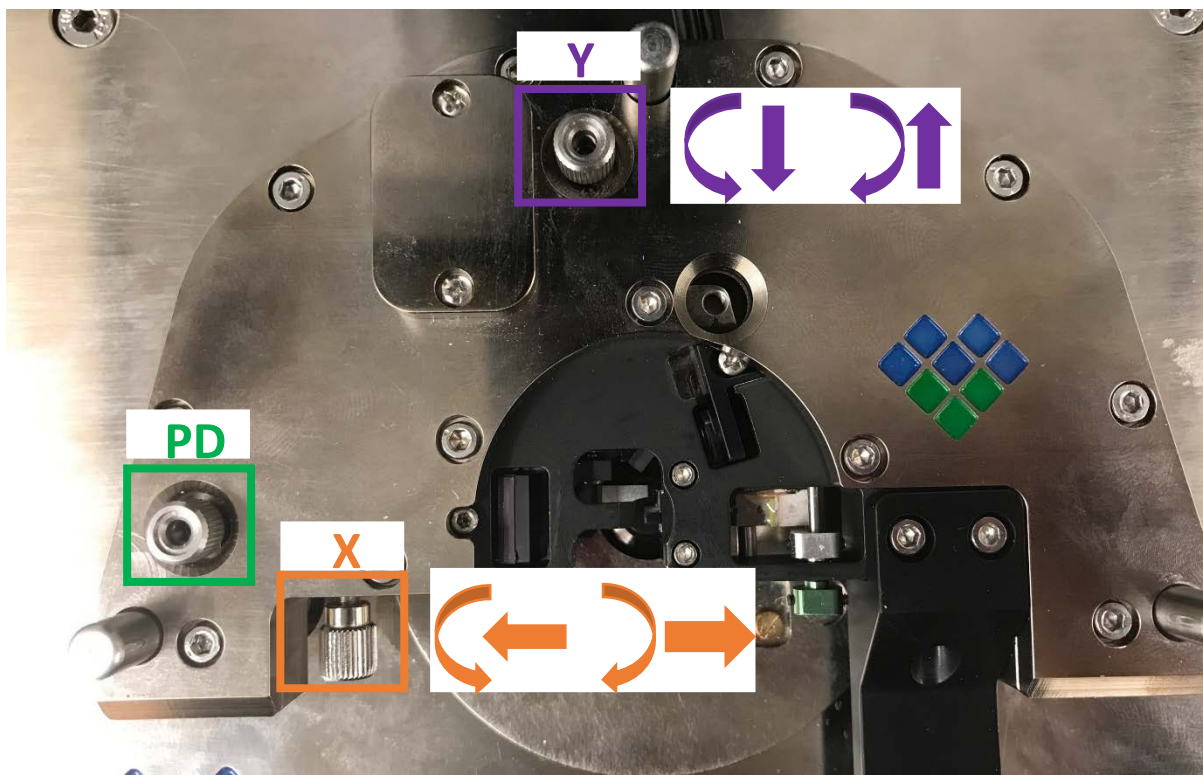
Any specifics that are not mentioned in this SOP can be found in the VistaScan Reference Manual on Molecular Vista's Website at <http://molecularvista.com/help>.

In addition, if there are sections you think can be improved, please let Jessica know at kongjy@uw.edu.

1. Flip the green notch and **load a tip**. It should fit squarely into the chip holder (blue box). Flip the green notch to secure.
2. **Place the AFM head on the stage**, making sure that the two front posts fit into the circular (yellow box) and rectangular (orange box) grooves on the head. Plug in the cable hanging off of the head.
3. **Open the latest version of VistaScan**. As of this writing it is 1.6.1b11. The steps below will walk through each tab.



4. **Sessions.** Change the User and Sample fields as necessary. For the software to register the changes, you must hit the Enter button. Click the "Browse..." button to change the directory to which your images will be saved. Under Feedback mode, select PiFM to take PiFM image or hyPIR to take hyperspectral images. Select Feedback 2nd mode, Detection 1st mode, sideband (default) as the PiFM mode.

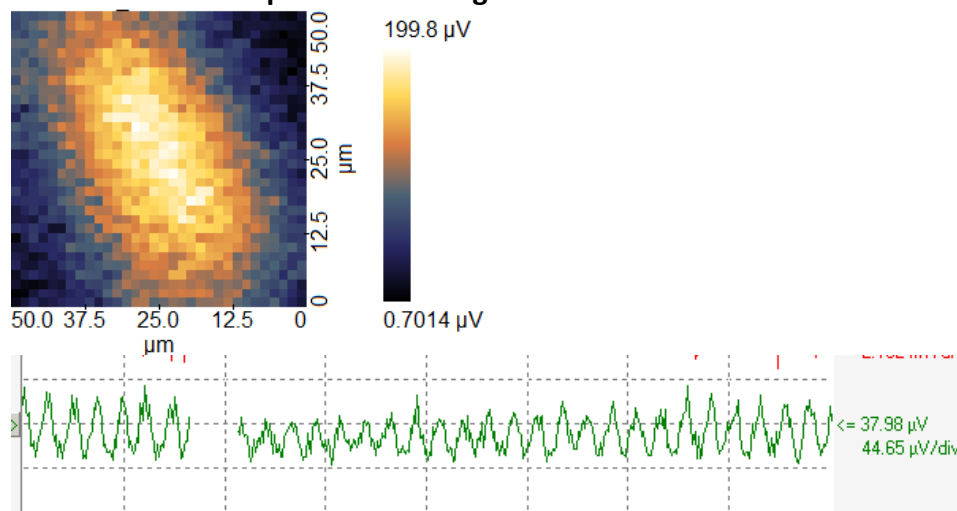


5. **AFM Setup.** There are two sections: Sensor Alignment and Frequency Sweep.
 1. *Sensor Alignment.*
 - a. The Total Intensity needs to be maximized and the Imbalance close to zero. *Do not adjust the photodiode (PD) knob until you have maximized the intensity.*
 - b. Adjust the position of the deflection laser by adjusting the X and Y knobs as necessary to maximize the total intensity. It should be around 950-1000 if you are using a platinum tip and around 1100 if you are using a gold tip.
 - c. Zero out the imbalance by adjusting the PD knob. Now you are ready to tune the tip!
 2. *Frequency Sweep.*
 - a. Press the Auto button to tune the tip. The drive amplitude can be adjusted but 1 nm is a good place to start. The drive voltage at 1 nm should be on the order of tens of mV. If it is several hundred mV, you can go through the steps listed in 5.1 again or reseal the tip. If the drive amplitude is still on the order of hundreds of mV, you might have a bad tip!

6. Approach/Scan: Aligning the Parabolic Mirror.

- a. Before looking at a sample of interest, make sure the parabolic mirror is aligned. Remove the head, flip it upside down (the three posts will serve as legs), and place it on a flat surface where it will not be knocked over.
- b. Place a glass coverslip onto the sample stage and secure it with the four pins. Use a flathead screwdriver to loosen and tighten the screws and a pair of tweezers to move and hold the pins as you secure them. Replace head. Don't forget to plug it in!
- c. Bring the tip as close to the surface of the glass as you feel comfortable by pressing the bottom button in the first column within the *Motors* section. This button has two arrows pointing at each other. Then, click the Approach button; the head will automatically lower such that the tip is on the surface. Pretty nice, right? The tip pictured in the Z Pizeo box will be green once the tip is on the surface.
- d. Once the tip is on the surface, select Parabolic XY from the dropdown menu next to Scan and then select "Yes" when prompted to change the resolution. A scan speed of 2 lines/sec and time constant (T. Const) of 10 ms should work nicely. Make sure the scan width is 50 μm so the entire range of the parabolic mirror is swept. Select the Laser Control tab and change the wavenumber to 1030 cm^{-1} (hit enter!); turn the laser on by clicking the Laser On button.
- e. Scan and keep an eye on a couple of things:
 - i. The PiFM channel in the Oscilloscope (green squiggle at the top of the box on the right hand side). The PiFM signal will oscillate if the laser is aligned to the tip.
 - ii. The PiFM image (click on the icon with four boxes at the top of the box in the middle panel). There will be an ellipse if the laser is aligned to the tip.

This is what the ellipse and PiFM signal should look like:

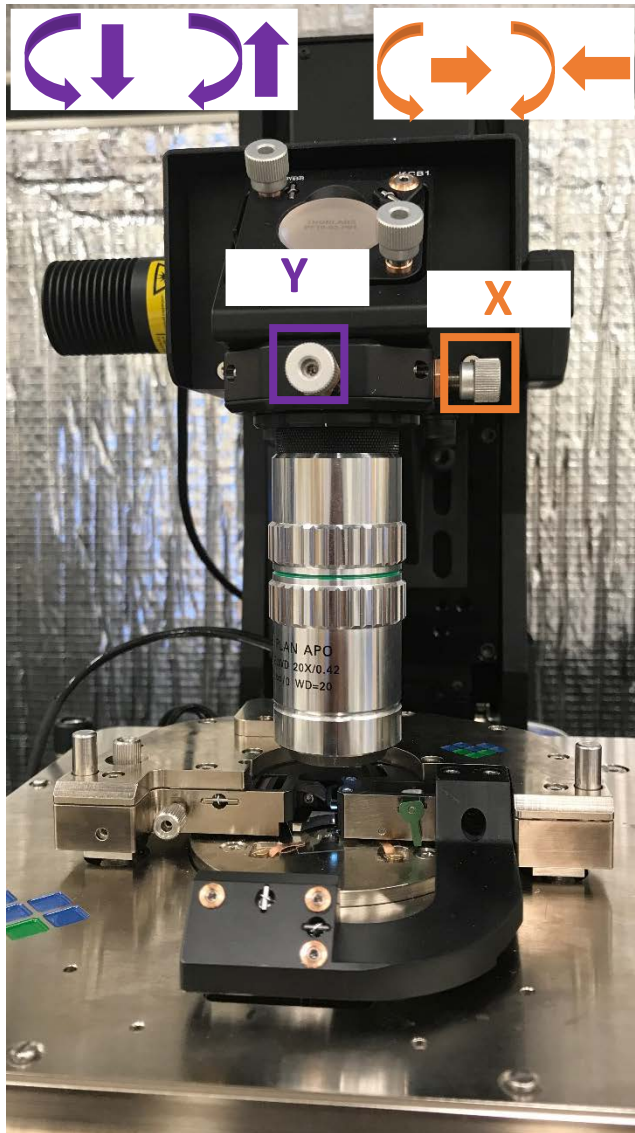


The actual value of the PiFM signal may vary depending on the laser intensity and pulse duration.

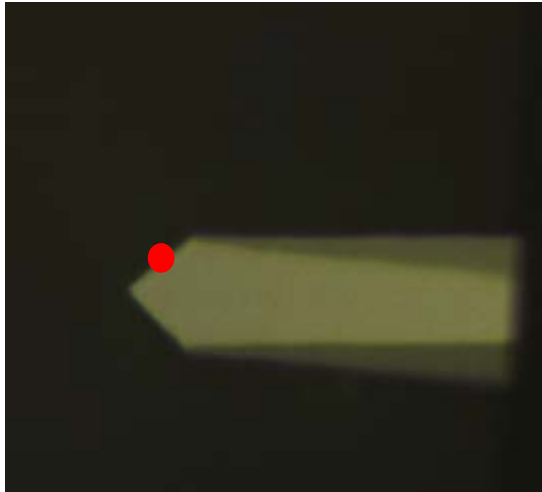
It's more likely than not that you'll have to adjust the position of the parabolic mirror. Below are couple possible scenarios and what to do:

1. You see part of an ellipse, but it is not entirely within the PiFM image. That's pretty good and means the parabolic mirror only needs to be adjusted minimally.
 - a. First, retract the tip under the Approach/Scan tab by clicking the up arrow in the Z Piezo box.
 - b. Under Coarse Movement, in the Head section, click Parabolic Mirror. ONLY click the arrows in the Horizontal section. These buttons adjust the position of the parabolic mirror in the XY-plane. The direction of the arrows correspond to the direction the image will move. So if you want to move your image to the left, click the left arrow. Start by a couple of clicks to in one direction to get a sense of how far the spot moves.
 - c. Engage the tip again by clicking the down arrow in the Z Piezo box under the Approach/Scan tab.
 - d. Scan Parabolic XY again. The spot should have moved. Move the parabolic mirror again as necessary to move the entire spot in the field of the PiFM channel.

2. The PiFM image is noisy and there is no bright glob. We'll need to use the camera and guide laser to move the laser onto the tip.
 - a. Click on the "Camera – Top View" button in the middle panel. Turn the LED on and lower the camera by clicking the down arrow in the Top Focus box under Coarse Movement. The buttons with two arrows moves the camera more quickly. Adjust the focus until the tip comes into view. To move the field of view, adjust the two knobs on the collar.



- b. After the tip is in view, go back to the Laser Control tab and turn on the Pointer by clicking Pointer On. You should see a red spot which is the guide laser.
- c. Under Coarse Movement, in the Head section, click Parabolic Mirror. ONLY click the arrows underneath Horizontal section. These buttons adjust the position of the parabolic mirror in the XY-plane. Position the guide laser on to the tip. Typically the laser is aligned to the tip when the guide laser is on the edge of the tip as diagrammed below.



- d. Under the Approach/Scan tab, click the down arrow to engage the tip again. Scan again and watch the oscilloscope and the PiFM image.
- e. Repeat d and e until you see an ellipse.
- f. Once the ellipse is in the field of view, right click in the center of it under the Four channel view and select “Move tip here.”
- g. Then switch the Scan to Parabolic YZ and repeat. This bright spot in the YZ channel is closer to a circle than an ellipse. If the spot is not in focus, you will need to click on the up and down arrows under Vertical and Horizontal (but not the left and right arrows under Horizontal). To move the spot along the y axis of the PiFM image, click on the arrows underneath the Vertical section. Clicking down will move the spot down. **The vertical buttons are very sensitive. Click only once before scanning in Parabolic XY again.** To move the spot along the x axis of the PiFM image, click on the arrows underneath the Horizontal section. To move the spot to the left, click the down button.

Note: Starting out, it’s a good idea to err on the side of caution and scan Parabolic XY (no, that is not a typo!) after clicking one button so the spot is never moved out of the field of view. Right click in the center of the spot in the XY and select “Move Tip Here.” Switch back to Parabolic YZ, rescan, right click in the center of the spot, and select “Move Tip Here.” The process of moving the parabolic mirror, right clicking, and selecting “Move Tip Here,” can be iterated and should end with moving the tip in the Parabolic XY scan.

7. Approach/Scan: Your Sample.

- a. The hard part is done! Glue your sample onto a glass coverslip with epoxy.
- b. While the epoxy dries, turn off the laser, raise the head with the first button in the first column under Motors. Make sure to raise it enough to account for your sample – you don’t want to crash a tip when you place the head back! Remove the glass coverslip and once your sample has dried and mount your sample.

- c. Lower head and click approach as before. Select “Sample” from the Scan drop down menu.

8. Spectroscopy.

- a. Turn the laser on and take a point spectrum to see what wavenumbers might give you interesting contrast. Refer to Molecular Vista’s page on Set Up, Acquire, and Correct IR-PiFM Spectra for technical detail.
(<http://molecularvista.com/set-acquire-correct-ir-pifm-spectra/>)

Quirk: If you take a point spectrum, the laser will be stay at the upper limit of the wavenumber range of your spectrum. You must click the wavenumber box and hit enter if you want to image at the last wavenumber you specified.

9. Approach/Scan: Your Sample.

- a. After selecting the wavenumber at which you want to image, engage the tip again by clicking the down arrow and scan. A few notes on scan parameters:
 - i. *Speed.* I’ve found that adjusting the scan speed such that the tip speed is about 5.0 $\mu\text{m/s}$ gives pretty nice images.
 - ii. *Parameters under z, Fastz, T. Cont tabs:* click “?” to reference Molecular Vista’s guide.

Other notes about scanning:

- a. Adjusting the scan speed, set point, integral gain (K_i) under z and Fastz, as well as the drive amplitude can increase the quality of your images.
- b. Adjusting the PiFM detection frequency can also increase the quality of your images. See Molecular Vista’s notes and video here:
<http://molecularvista.com/help/1-6-reference-manual/pifm-tuner/>