FIGURE 1. EXPERIMENTAL SETUP. THE RED ARROWS INDICATE THE BEAM PATH. THE PARABOLIC MIRROR (PBM) HAS MICROMETER ADJUSTORS IN 3 DIMENSIONS. PLEASE USE ONLY THE FINE ADJUSTORS. THE MCT DETECTOR HAS A GOLD CAP WHICH NEEDS TO BE REMOVED FOR FILLING WITH LIQUID NITROGEN.

Quick Start Procedure

Abbreviated steps for collecting SINS data are shown below. For more information about any step, please click on the bookmark to navigate to the appropriate section of the document.

1) <u>Create User directory</u>
2) <u>Cool MCT detector</u>
3) <u>Turn on MCT detector</u>
4) <u>Configure Omnic software for Measurements</u>
5) <u>Open Zurich lock-in software</u>
6) <u>Open Nanodrive software</u>
7) <u>Load the sample</u>
8) <u>Align AFM laser</u>
9) <u>Autotune AFM cantilever</u>
10) <u>Change setpoint to ~ 3.7 (assuming 5 V withdrawn tapping amplitude)</u>
11) <u>Engage tip</u>
12) <u>Open Hardware Access</u> (only required when first opening Nanodrive)
13) <u>Check Zurich lock-in parameters</u>
14) <u>Align IR beam onto tip</u>
15) <u>Perform AFM scan</u>
16) <u>Check Omnic parameters</u>
17) <u>Collect spectra</u>
18) <u>Withdraw tip</u>
19) <u>Turn off MCT detector</u>
      Advanced Users Only
20) *<u>Tip change</u>*
21) *<u>Optical alignment</u>*
22) *<u>Programming measurement locations</u>* (series of points, linescan, area scan)

<u>Troubleshooting</u>
No lock-in signal
No interferogram
Spectrometer not scanning
AFM navigator has blue screen
AFM sample stage tilts forward
Trouble finding alignment laser after tip change
Alignment laser walks or jumps
Sample drift
Tip not making contact
Sinusoidal oscillations in spectra

MCT Detector

The detector should be filled with liquid nitrogen using a funnel and the 4L dewar provided. If there is no nitrogen, the dewar can be filled at the user liquid nitrogen station on the main floor of the ALS, near building 15 and across from beamline 2.1. If you haven't received liquid nitrogen training from either Hans or Stephanie, please wait for the Beamline Scientists to arrive before working with any cryogens.
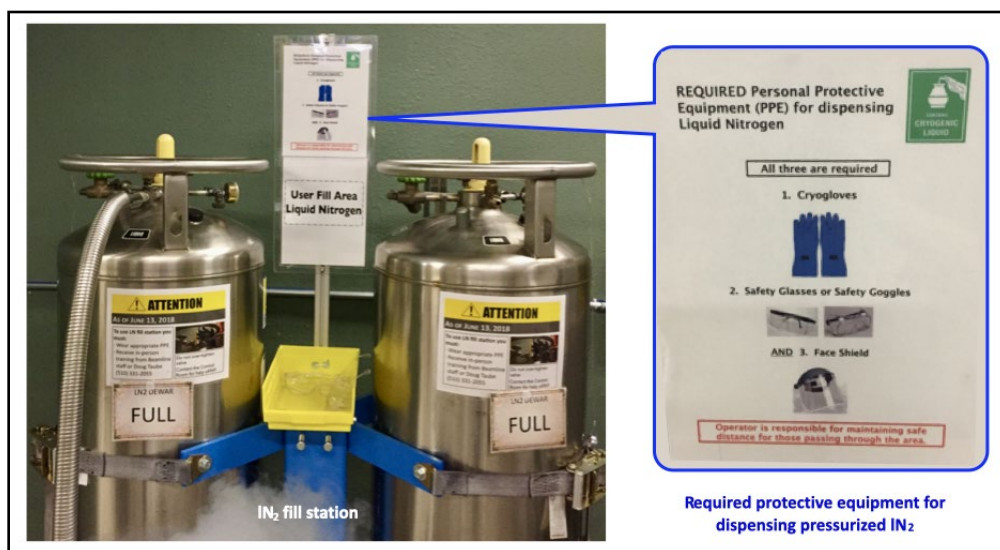

FIGURE 3. LIQUID NITROGEN FILL STATION AND PPE REQUIREMENTS. USERS MUST BE TRAINED BEFORE OPERATING THE FILL STATION AND WEAR THE REQUIRED PROTECTIVE EQUIPMENT. CRYOGLOVES, SAFETY GLASSES UNDER FACE SHEILDS ARE REQUIRED AT THE FILL STATION.
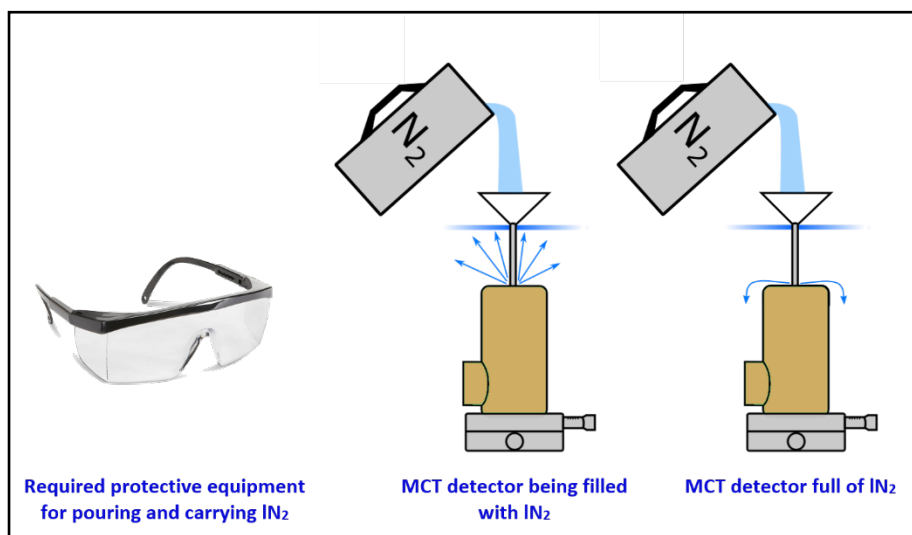

FIGURE 4. FILLING THE MCT DETECTOR WITH LIQUID NITROGEN. SAFETY GLASSES ARE REQUIRED. REMOVE THE GOLD CAP FROM THE DETECTOR AND INSERT THE FUNNEL. SLOWLY POUR THE NITROGEN AND FILL THE DETECTOR UNTIL LIQUID JUST STARTS OVERFLOWING FROM THE RESERVOIR.

Once the detector is cold (about 10 minutes after filling with lN$_2$), the detector can be turned on using the round power button. The output button located on the left-hand side of the power supply (underneath the table) also needs to be pressed. The power supply will automatically recall the correct current and voltage settings.

FIGURE 5. POWER SUPPLY FOR THE MCT DETECTOR. THE SUPPLY AUTOMATICALLY RECALLS THE APPROPRIATE SETTINGS: TURN ON THE POWER WITH THE LOWER BUTTON (INDICATED BY LOWER RED ARROW), THEN ENABLE THE OUTPUT BY PUSHING THE BUTTON ON THE UPPER LEFT (UPPER RED ARROW).

The detector will stay cold approximately 10-12 hours, at which point it will need to be refilled with liquid nitrogen. It is recommended that Users keep a log of the detector fill times.

Please turn off the power supply when you finish measuring for the day/night.

Nanodrive AFM Software

Open the Nanodrive software version 8.05. Verify Tapping SINS is selected in the system configuration as shown in Figure 6. Click on the green *Load Experiment* button in the lower right-hand corner of the window.

This will launch the main Nanodrive window (Figure 7) and also open up the Navigator window, which provides a top-down optical image of the tip and sample.

Users can drag the components of the AFM signal within the *Nanodrive* window to display the relevant signals in the four large squares. Note: BNC 1 is the near-field amplitude signal from the lock-in, typically configured as
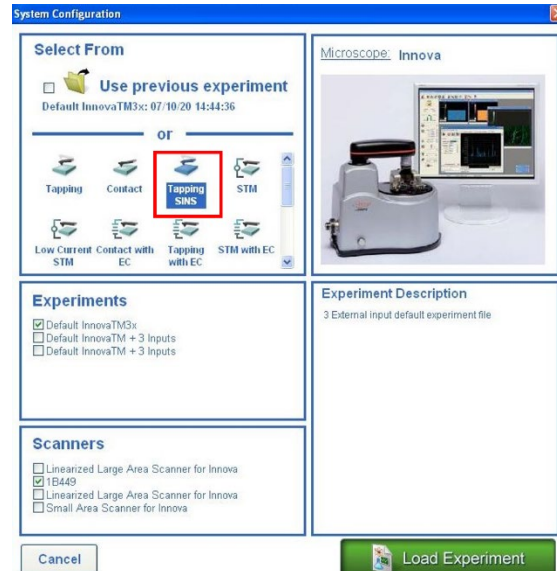

FIGURE 6. SYSTEM CONFIGURATION. VERIFY TAPPING SINS IS SELECTED AND THEN LOAD EXPERIMENT.
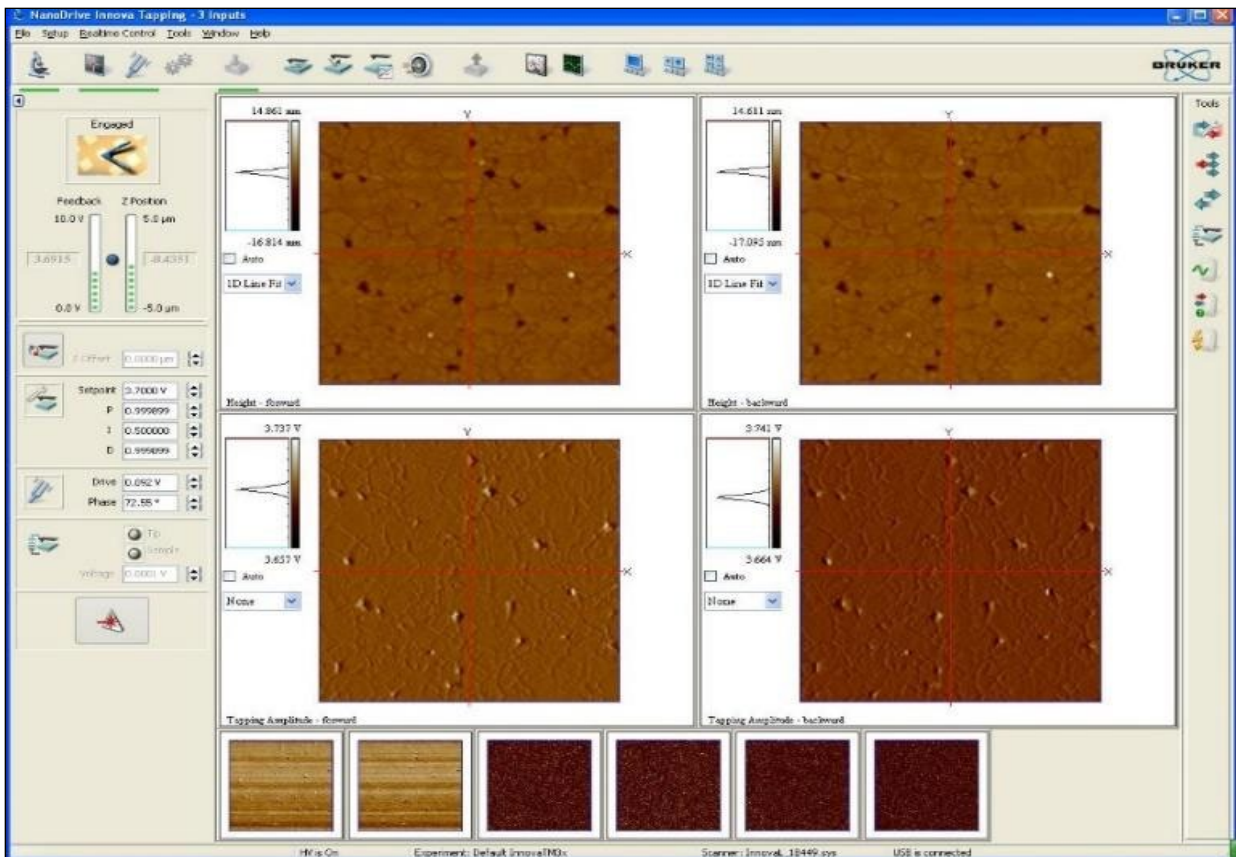

FIGURE 7. NANODRIVE EXPERIMENT WINDOW. THE BROADBAND IR NEAR-FIELD AMPLITUDE RESPONSE IS IMPORTED INTO THIS WINDOW FROM THE ZURICH LOCK-IN AS BNC1 (DEMOD2 OR 2ND HARMONIC).

Demod2 (2nd harmonic). BNC 2 is used for triggering line scans, and generally isn't used for imaging.

Within each of the four squares, there is a drop-down option to perform different types of leveling on the displayed response. Leveling can be used on the AFM signals such as the height profile, but _should not_ be applied to the near-field response in the BNC 1 window.

⚠️ Any leveling applied to the AFM images is saved when the data is saved.

In addition to the _Nanodrive_ window, the Navigator window will also appear when the experiment is loaded. This window displays the view from the optical microscope (Figure 8). The illumination slider can be used to adjust the brightness of the internal microscope light, and the (digital) zoom can be used to increase or decrease the field of view.

In a typical alignment, the cantilever and chip edge are visible, with the cantilever apex in the cross-hairs. It is helpful to keep in mind that the optical microscope and cantilever/chip are fixed and only the sample stage moves during operation.

Returning to the same tip/cantilever position will _significantly_ speed up alignment of the IR light.
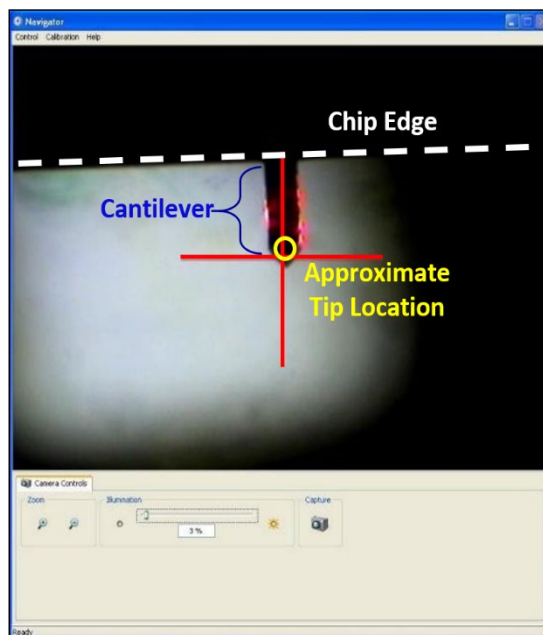

FIGURE 8. NAVIGATOR WINDOW. THIS WINDOW DISPLAYS THE OPTICAL MICROSCOPE IMAGE OF THE CANTILEVER AND SAMPLE SURFACE. WITH THE ILLUMINATION TURNED DOWN.

Loading your Sample

Unplug the electrical connection and then remove the AFM head from the three-point mount by lifting up slightly and pulling back, being careful not to bump the objective. Place the AFM head upside down (as shown in the lower right of Figure 9) to avoid misaligning or damaging the tip.
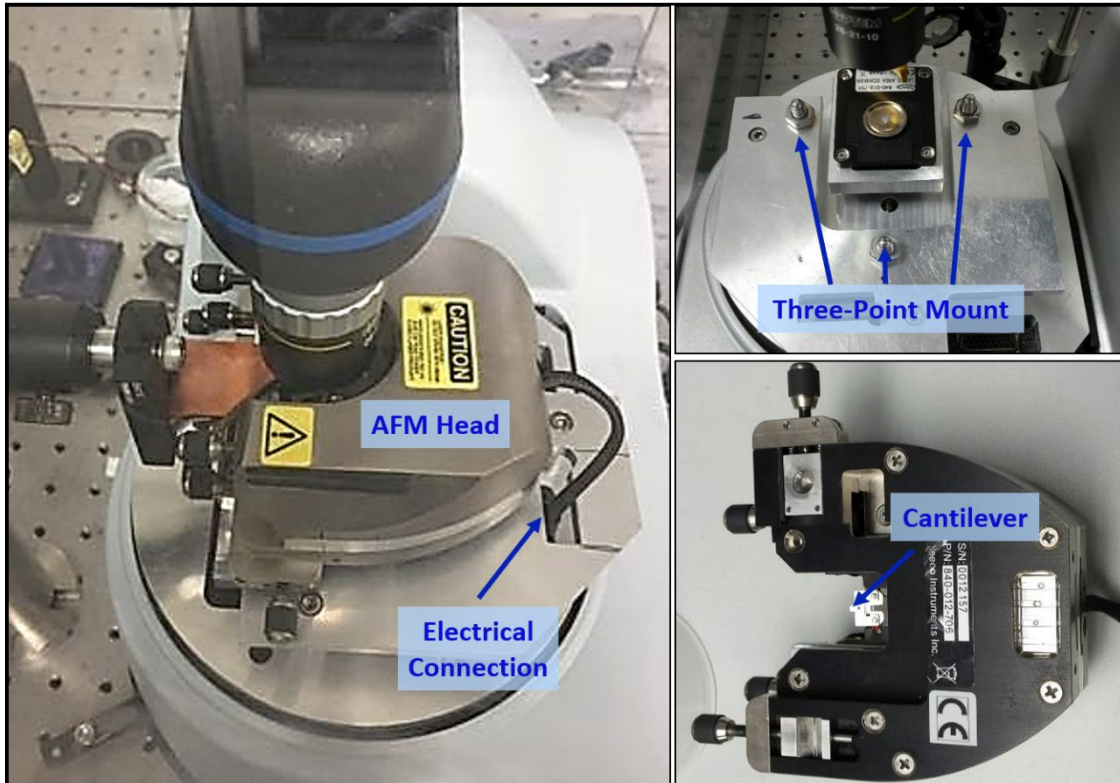
FIGURE 9. AFM HEAD AND MOUNT. LEFT: SIDE-VEW OF THE AFM HEAD IN PLACE ON THE MICROSCOPE BASE. UPPER RIGHT: THE BASE AFTER THE HEAD HAS BEEN REMOVED. THE THREE-POINT MOUNT IS INDICATED WITH ARROWS. LOWER RIGHT: THE AFM HEAD UPSIDE-DOWN WITH THE CANTILEVER POSITION INDICATED BY THE BLUE ARROW.

Choose *Setup >> Engage* to open the engage menu. This opens a small window that allows you to withdraw the tip (top left button, red arrow pointing up), engage the tip (middle left button, green arrow pointing down) and manually adjust the sample z-direction (bottom, left buttons). Note that the *arrows refer to the relative motion of the tip*, even though the sample is actually moving (i.e. arrow up causes sample to move down, effectively raising tip.)

Attach a sample to the mounting block. Double-sided tape works but can lead to additional thermal drift. Carbon tape and copper tape appear to perform better. Fingernail polish, silver paste, and glues work best, but are harder to remove from your sample.

Place the mounted sample on the piezo stage, being careful not to move or bump the microscope objective.

With the AFM head still removed, press the *Tip up/Tip Down* buttons (lower buttons) to find the sample focus in the *Navigator* window. Right-clicking the buttons will allow you to change the step size.

Manually move the sample to an area of interest using your fingers, being careful not to bump the AFM objective or the parabolic mirror. Note that the piezo scanner only has a 100 μm x 100 μm scan range.
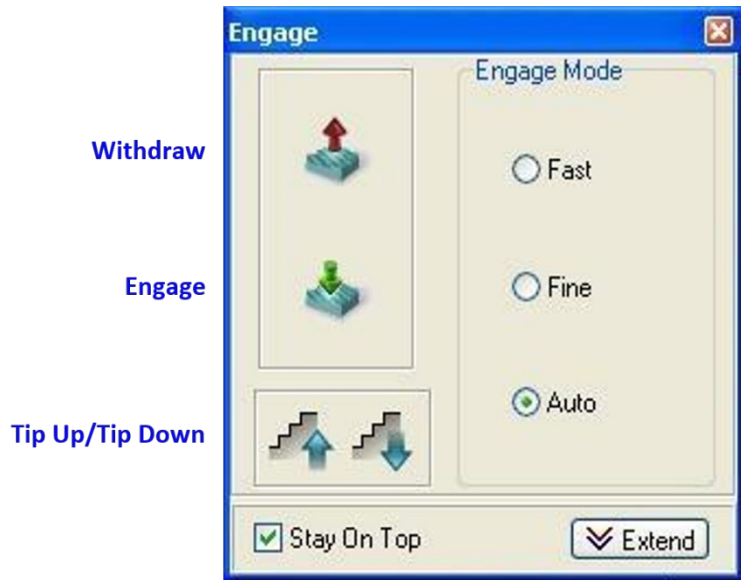

FIGURE 10. ENGAGE WINDOW. THE ARROWS REFER TO THE RELATIVE MOTION OF THE TIP. REMEMBER THAT THE SAMPLE STAGE IS ACTUALLY MOVING.

After identifying a region of interest, lower sample by pressing the *Withdraw* tip button (red up arrow), such that tip is withdrawn by at least 250 μm.

Replace the AFM head on the three-point mount, being careful not to bump sample or objective. Carefully reconnect the electrical plug.

⚠️ *Do not* use the Tip Up/Tip Down buttons when the AFM head is on. You can easily crash your tip.

If the laser alignment and cantilever tuning are optimized, you can push the *Engage* button (green arrow, down) to automatically place the tip in contact with the sample. Otherwise, continue onto the laser alignment and cantilever tuning procedures.

Laser Alignment

Click on the *Laser Alignment* button in the upper toolbar ribbon of the *Nanodrive* window. This will open up the laser alignment window as shown below in Figure 11. In the *Navigator* window, you should see the red laser on the AFM tip.

FIGURE 11. DEFLECTION LASER ALIGNMENT. LEFT: POSITION SENSITIVE DETECTOR (PSD) FOUR QUADRANT SIGNAL IN THE LASER ALIGNMENT WINDOW. RIGHT: OPTICAL MICROSCOPE IMAGE OF CANTILEVER IN THE NAVIGATOR WINDOW.
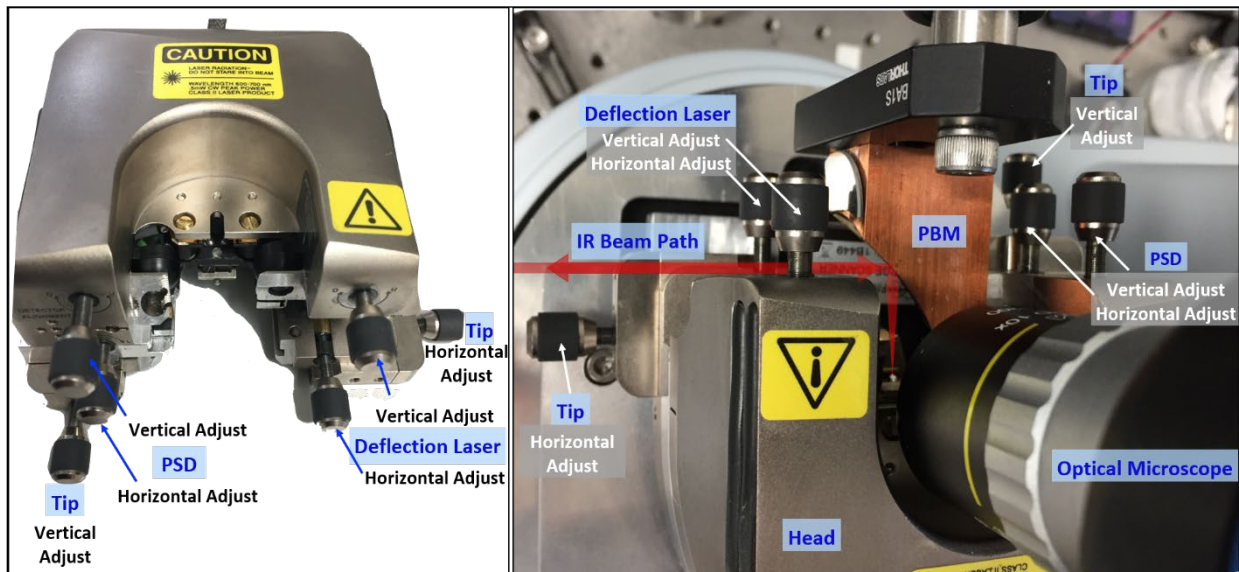

FIGURE 12. AFM HEAD POSITIONING KNOBS. LEFT: AFM HEAD AS VIEWED FROM THE DIRECTION OF THE PARABOLIC MIRROR. RIGHT: THE AFM HEAD IN PLACE. THE LEFT/RIGHT DIRECTIONS LISTED BELOW REFER TO THE AFM HEAD IN PLACE.

The general procedure for aligning the deflection laser is to use the vertical and horizontal knobs to adjust the laser position on the cantilever while maximizing the Sum signal, and then using the position senstivie detector (PSD) knobs to center the pink circle on the cross hairs.

(1) Using the laser alignment knob (top left knob when facing optical table; Figure 12 right image), adjust the vertical position of the laser such that it is near the bottom of the tip cantilever, but fully on the cantilever.

(2) Using the laser alignment knob (bottom left knob when facing optical table, Figure 12, right image), adjust the horizontal position of the laser to maximize the Sum signal number at the bottom of the laser alignment window.

For PtSi tips: Sum signal ~ 1.3 - 1.5 V

For gold tips: Sum signal ~ 1.5 - 1.7 V

(3) Using the detector alignment knobs (upper right knob in Figure 12, right image), center the pink circle onto the cross hairs in the laser alignment window. Aim for a value less than |.2| V in both the vertical and horizontal.

- The upper right knob adjusts the vertical position

- The middle right knob adjusts the horizontal position

For both detector knobs, if the number is positive, screw in/clockwise; if the number is negative, screw out/counter-clockwise.

⚠ Use caution when adjusting the horizontal laser position as turning the knob too far screw in/clockwise will break it!

⚠ Be careful to not adjust the bottom left and right knobs (tip alignment). This moves the tip and will cause misalignment.

Note: When the AFM head is first plugged in, the horizontal position typically drifts in the negative direction for several minutes. It is usually helpful to compensate for the drift by aligning slightly off center in the positive horizontal direction.

If the laser alignment drifts completely out of the window when no changes are made to the system, or if small changes to the laser alignment produce erratic and irreproducible changes in the laser position, notify a Beamline Scientist immediately.

Cantilever Tuning

After aligning the deflection laser, click on the *Cantilever Tuning* button in the upper toolbar ribbon of the *Nanodrive* window, opening the tuning window shown in Figure 13.
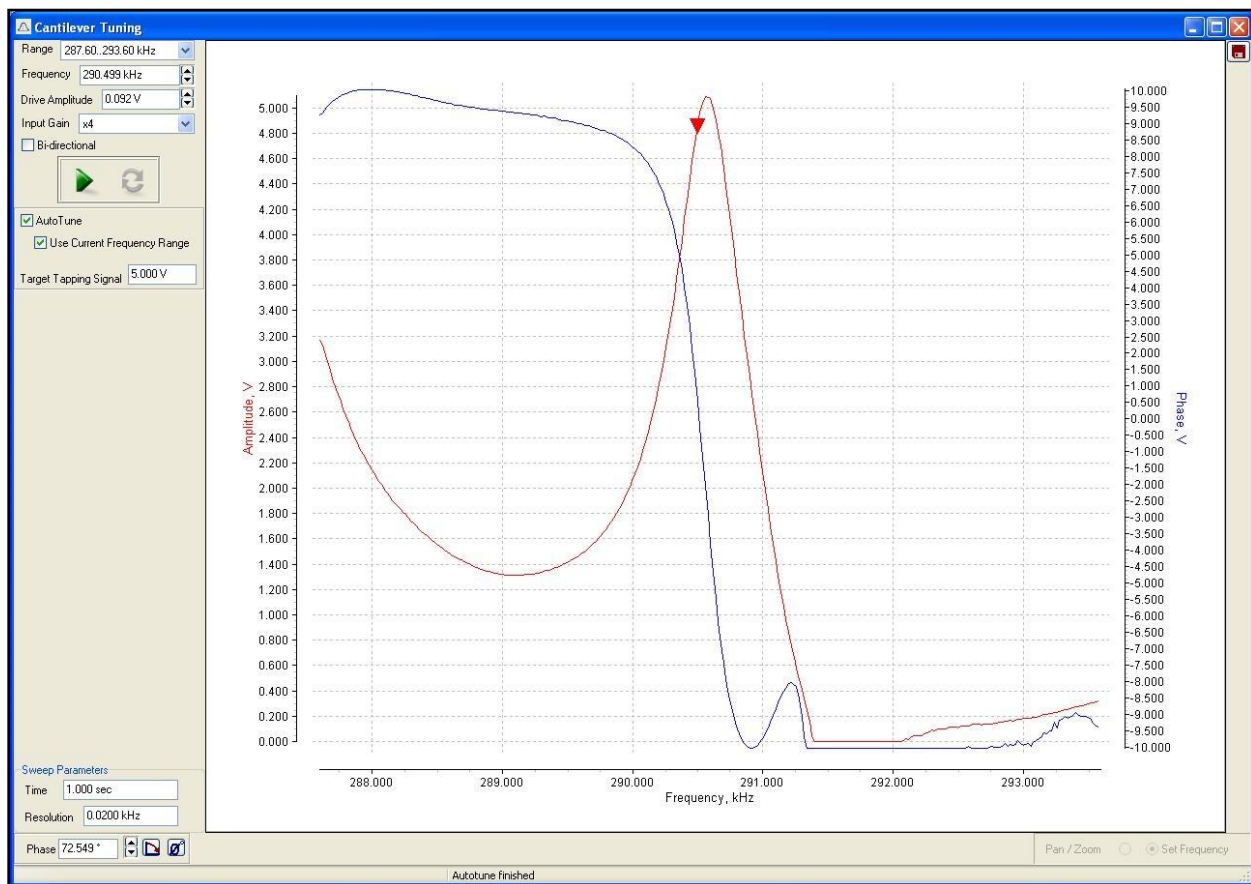

FIGURE 13. CANTILEVER TUNING WINDOW. THIS FIGURE SHOWS THE RESULTING TUNING CURVE FOR A STANDARD TIP.

For a new tip, use the following settings:

Range*: 0-1000 kHz*   Autotune box checked*.*

Input Gain*: x4*   Target Tapping Signal*: 5.0 V*

Push the green arrow to begin the autotune process. The amplitude (red) trace should have a nice resonant peak similar to image above with the peak near 5 V, and the phase (blue) should look sigmoidal and range from 10 to -10 V, going through zero at the frequency position of red arrow. The normal resonance frequency for the tips we use is 250 - 310 kHz. The drive amplitude is typically between 0.02 - 0.1 V.

<u>Return to Quick Start Procedure</u>

Setpoint

The setpoint is the primary feedback mechanism for the AFM. A lower setpoint brings the sample closer to the tip. It should be set to less than the free oscillation of the cantilever, which is nominally adjusted to 5 V by the Autotune procedure. The Autotune procedure automatically selects a setpoint, but it is typically too low (too close to the sample surface and more likely to damage the tip).

Change the setpoint to ~ 3.7 V, which is 75% of the free (withdrawn) tapping amplitude (nominally adjusted to 5 V). Depending on the tip and sample, you may need to further adjust the setpoint up or down, but this is a typical value and good place to start.

Press the *Engage* button (green arrow in the Engage window, Figure 10) in order to AutoEngage the AFM tip with the sample.

Hint: If you are engaged on your sample, you can change the setpoint to 10 V. This will effectively cause the sample to lower to its minimum value and disengage from the tip by ~3 μm. This can be convenient if you want to put a lid on the N$_2$ purge boxes or adjust something without losing your location on the sample. Returning the setpoint to ~3.7 V will re-engage the tip with the sample.
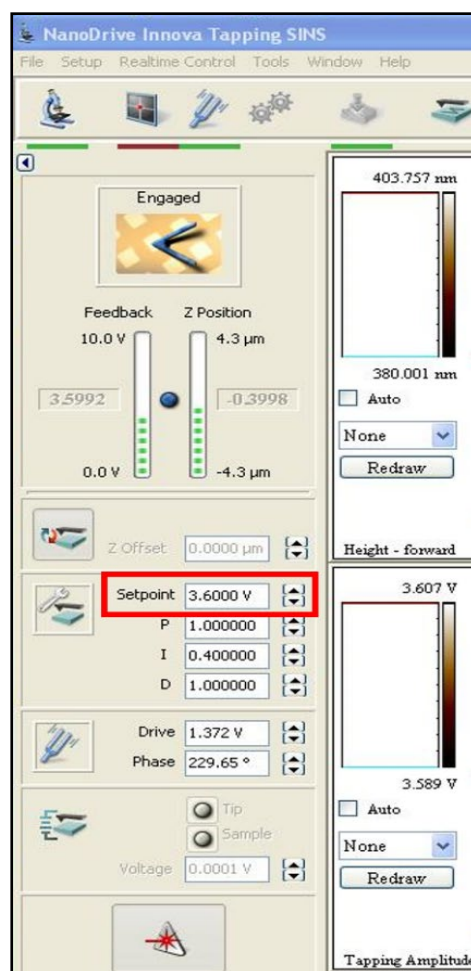

FIGURE 14. SETPOINT VALUE. THE RED BOX INDICATES THE SETPOINT VALUE IN THE NANODRIVE WINDOW.

AFM Scanning Control

Once the AFM tip is engaged, the system will beep and the *Scanning Control* window will automatically open.

If the AFM tip is engaged properly, the Feedback Monitor on the main *Nanodrive* window should be stable at the chosen setpoint. Likewise, the Z-position monitor should be centered between the +/- 5 μm range. It is good practice to periodically look at these monitors during acquisition to ensure that the feedback control is working properly.

Note: if the feedback monitor indicates the tapping amplitude is well below the setpoint or the Z position monitor begins to exceed +/- 3 μm, it is generally best to withdraw the tip and re-engage. If the tip still isn't tracking properly, adjust the gain as described below.

The gain (of the feedback loop) is a multiplier applied to the error signal to generate the feedback signal to the scanner. These parameters (described in PID settings below) will likely need to be optimized for each set of scans. Higher gain values mean that the feedback loop is more responsive to changes in deflection of the cantilever.

FIGURE 15. SETPOINT AND FEEDBACK CONTROL. THE UPPER-LEFT OF THE NANODRIVE WINDOW HAS ACTIVE FEEDBACK AND Z POSITION MONITORS.

PID settings

The proportional feedback signal (P) is based on the difference between the current signal and the target.

The integral feedback signal (I) is based on the sum of previous errors (this method will correct a continuing set of errors too small to be corrected by other feedback methods).

The derivative feedback signal (D) is based on the difference between the current error signal and the preceding error signal.

The gains are multipliers for each of these feedback signals but the units for gain are arbitrary. In most cases, the integral gain value has the largest effect in optimizing feedback behavior in scanning probe microscopy.

> Gain too high: ripples or oscillations will appear in the image

> Gain too low: height doesn't track the topography

Set the gain by beginning with the default PID values (P=1.0, I = 0.3, D = 1.0). Gradually increase the value until the system begins to show oscillation and reduce the gain until the oscillation ceases. An acceptable gain setting usually will lie within a range of settings rather than at a single value.
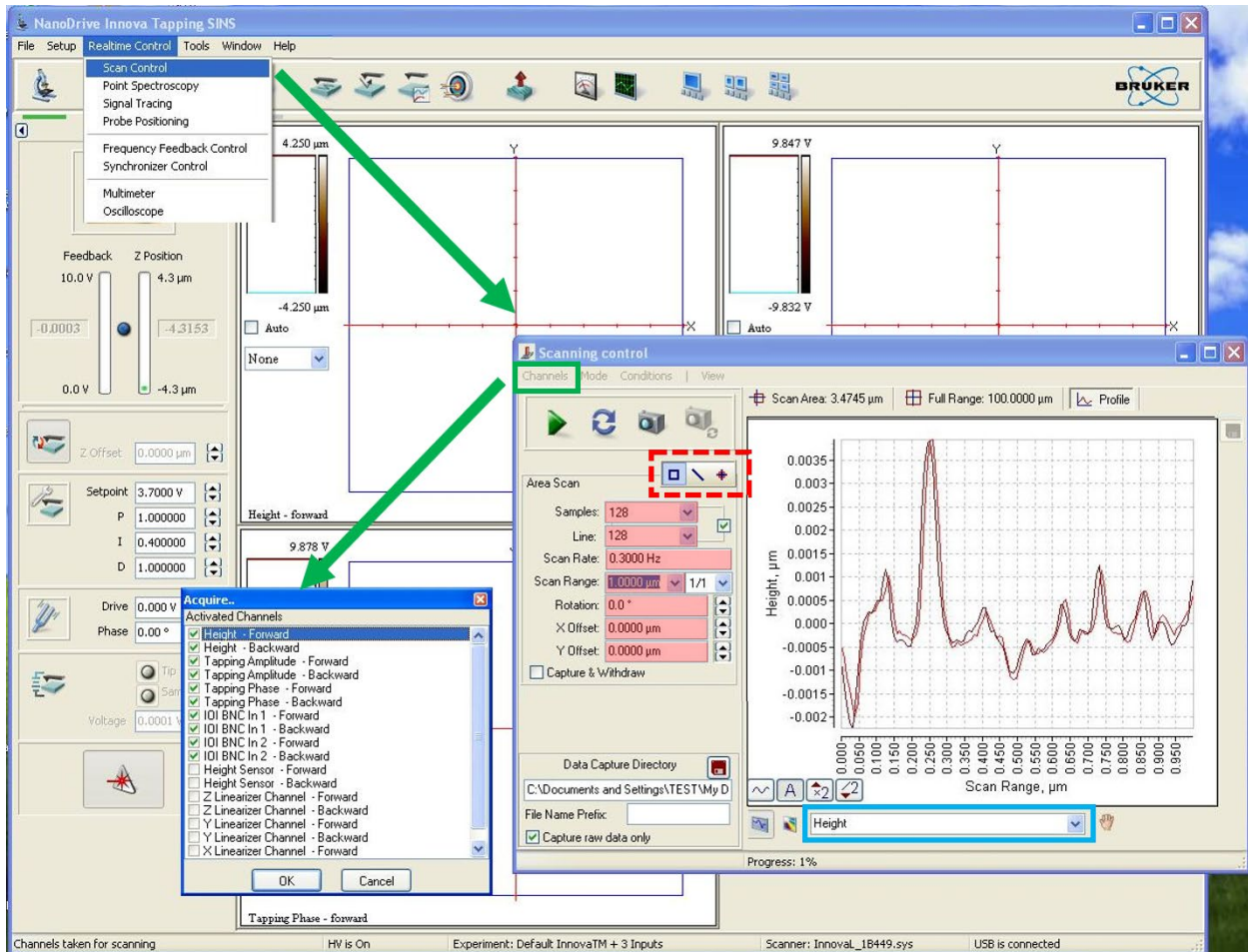
Scan Control Setup

FIGURE 16. CONFIGURING SCAN CONTROL. CONFIGURE THE NECESSARY CHANNELS FOR SINS AS SHOWN IN THE "ACQUIRE" BOX. THE DASHED RED RECTANGLE INDICATES WHERE TO SWITCH BETWEEN AREA, LINE, AND POINT SPECTROSCOPY SCANS. THE SCAN SETTINGS ARE HIGHLIGHTED IN RED. TO VERIFY THE HEIGHT IS TRACKING PROPERLY, CLICK THE PROFILE TAB AND SELECT "HEIGHT" AS THE CHANNEL TO DISPLAY. FORWARD AND BACKWARD TRACES SHOULD BE NEARLY IDENTICAL FOR PROPER OPERATION.

Select channels (*only necessary when software first opened*) by clicking the *Channels* button in the *Scan Control* window shown in Figure 16.

Select the first 9 channels for typical SINS operation: *Height* (forward/backward), *Tapping Amplitude* (forward/backward), *Tapping Phase* (forward/backward), and *BNC 1* (forward/backward).

The *Height channel* gives the *topography*.

The *Tapping Amplitude* shows the tapping amplitude, which should nominally be at the setpoint. However, it will change at abrupt height changes as the feedback control adjusts, making it a good *highlight for edges*.

The *Tapping Phase* shows the mechanical phase between the drive and response of the tip. It can be an *indicator of different elastic properties of the sample* and thus indicate changes in chemical identity.

BNC 1 and *BNC 2* are external inputs, typically from the Zurich Lock-in Aux 1 (Amplitude), and Aux 2, respectively. They show the *broadband infrared response* of a sample and the outputs can be adjusted with the ZI software. *BNC 2* is used for triggering linescans, not for typical near-field imaging.

Scanning Parameters (highlighted in Figure 16):

Samples: number of pixels (columns) per line (min. = 16x16 pixels, max = 1024x1024 pixels).
Lines: number of rows (can be set different than number of samples by unclicking checkbox)
Scan rate: sets the number of lines scanned per second in the x direction (min. = 0.1 Hz, max = 100.0 Hz). Usually ~.3 Hz for scan ranges < 10 μm. For larger scan areas, the scan rate should be lowered to avoid tip damage.
Aspect Ratio: Typically 1:1, but can be changed to rectangular scan areas.
Scan Range: size of the scan along one side of a square. The max range of the piezo is 100 x 100 μm.
Offsets: Denotes the center of the scan relative to the center of the full x-y scan range.
Rotation: combines x and y drive voltages such that the piezo scans the sample at variable x –y angles (scanning angle 0 to 360).

Suggestions for setting scanning parameters:

Generally, scan size should be selected to be greater than the lateral resolution multiplied by the number of data points per scan line, *i.e.*, the spacing between the pixels should be larger or comparable in size to the area covered by each data point. Selecting a smaller scan size will result in adjacent data points containing redundant information.

The scan rate can be adjusted while acquiring an image. In general, as the scan size is increased, the scanning velocity also increases (a longer line is scanned at the same scan rate, so the tip travels faster over the sample). Large scan sizes usually are better imaged by decreasing the scan rate in order to decrease the tip to sample scanning velocity. Slower scan rates give better resolution because the feedback system has time to respond, while faster scan rates save time. If the scan rate is too fast, the feedback loop may not respond appropriately to changes in height. As a result, the image may

appear smeared (poor surface tracking) or the tip may crash into protrusions on the surface, possibly damaging the tip and/or sample.

In the upper left-hand region of the scanning window, there is a square, a line, and a circle shown in a dashed rectangle in Figure 16.

> *Square*: selects area scanning
>
> *Line:* selects line scanning
>
> *Circle:* selects point positioning

In the top region of the scanning window, there are three tabs:

> *Scan Area:* shows the range of the current scan area (will be black when opening the software or any parameters are changed)
>
> *Full Range*: shows the full range of the piezo. A white box is displayed that shows the current scan area. This can be resized and repositioned to change the location and size of the scan.
>
> *Profile*: this will show individual lines during an area scan and can be used to fine tune the scanning parameters.

To perform an area scan, make sure that the square is highlighted and the appropriate parameters are selected. Push the green arrow in the upper left corner to start the AFM scan. To stop the scan, push the green arrow again. Images of the appropriate channels will build line by line in the main scanning window as the AFM scans.

To view individual lines in real time, select the profile tab in the upper right corner of the scanning window. The forward and backward height traces should reproduce each other, as shown in Figure 16. If not, check that the forward and backward traces have the same line correction in the image windows. Then try reducing the setpoint by .1 increments until the two traces are tracking each other.

Once the scan is finished, save the data by choosing *File >> Save Data* in the main *Nanodrive* window.   You can drag an image into scan control window and then use the point tool to position the probe a feature of interest.

Open Hardware Access

In order to select the tapping frequency input from the AFM, adjust the near-field signal, or acquire near-field data, you must first configure the *Nanodrive* software appropriately using Open Hardware Access: in the *Nanodrive* software, select from the upper menu: *Tools >> Open HW Access.*

A warning statement will appear; select *Yes* to continue. A new menu of icons will appear on the right side of the *Nanodrive* window, shown in the image on the right.

Click on the Innova Interface Board Control (second icon from bottom, circled in blue). The *IOI Board: recognized* window will open.

- For the Out 1 channel under Out BNCs, select *T-B fast signal*. This will send the tapping frequency of the AFM to the lock-in.

When finished, close the window by clicking the red x in the upper right of the window.
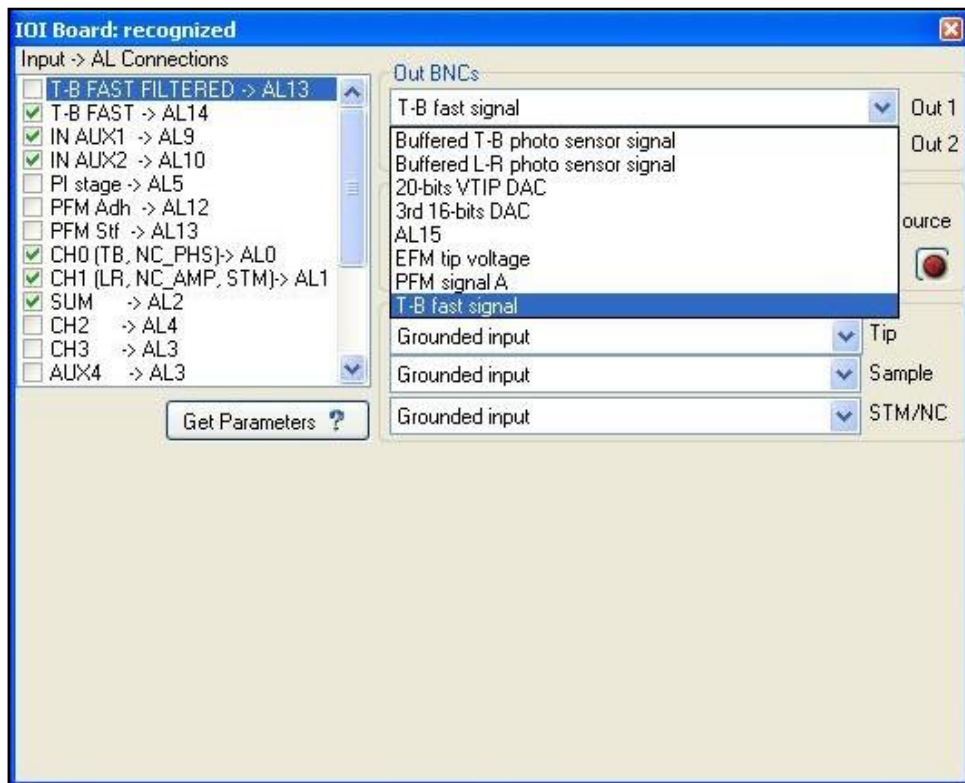
FIGURE 17. IOI BOARD. SETTINGS FOR THE INNOVA INTERFACE BOARD CONTROL WHICH SENDS THE AFM TAPPING FREQUENCY (REFERENCE FREQUENCY FOR DETECTION) TO THE LOCK-IN.

Zurich Lock-in

The *ziControl* software controls the lock-in amplifier settings and outputs. The settings are usually stored in memory and will retain their previous values when the program is reopened. Occasionally, these values will be restored to their defaults after a computer crash or communication error. The following gives screenshots of the *ziControl* software and a brief explanation of the more important parameters.

Lock-in MF Tab:
The Lock-in MF tab contains the primary parameters for the lock-in detection. A default configuration file "SINS Config Default.zicfg" for the Lock-in is available on the AFM computer desktop; navigate to the *Save* tab and click the *Load Settings* button to load the default settings. Alternatively, adjusting the parameters to what is displayed in Figure 18 is a good starting point. More details are listed below to fine tune the parameters.
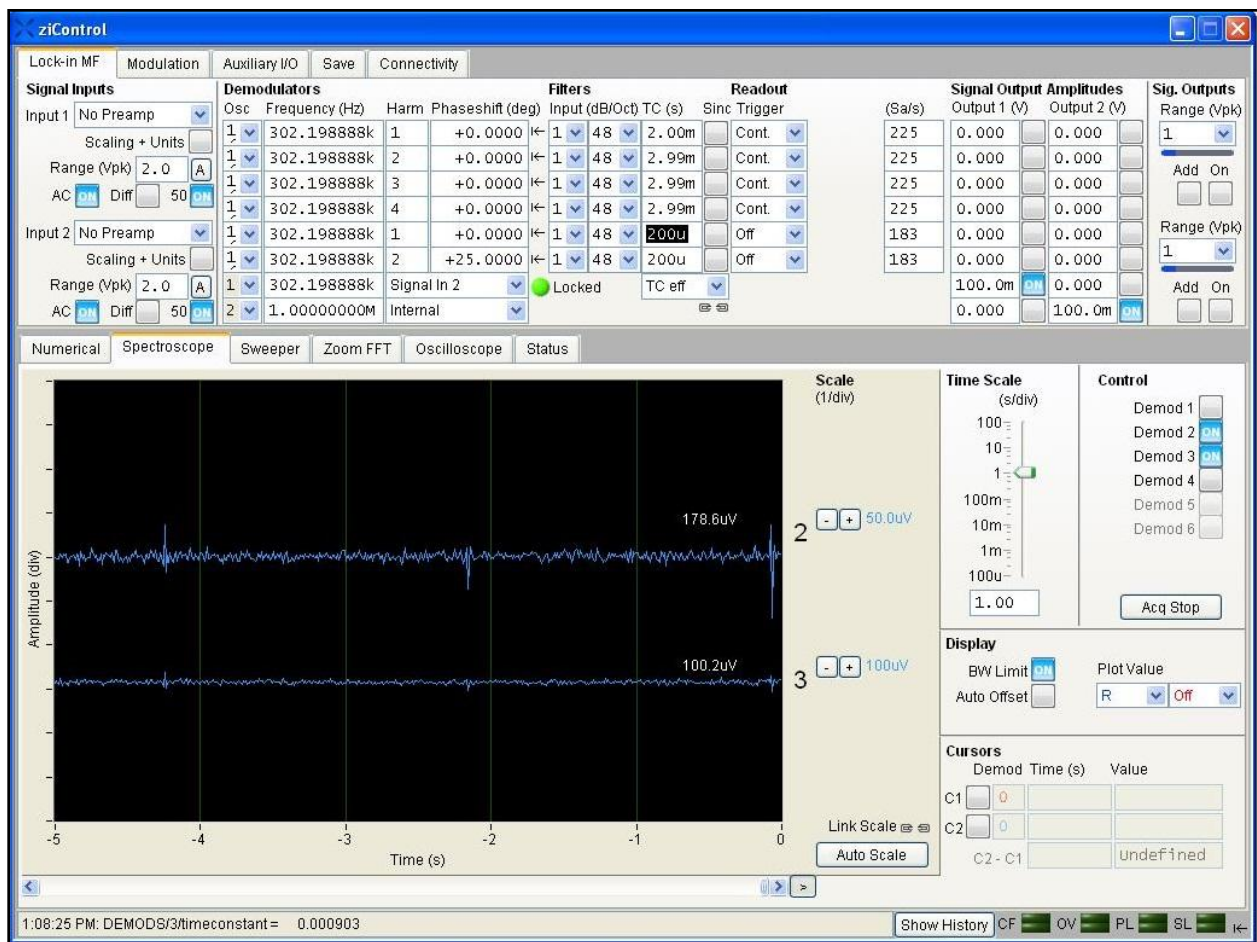

FIGURE 18. LOCK-IN SETTINGS. TYPICAL PARAMETERS FOR OPERATION.

Signal Inputs:

Input 1: This is the detector response. Check that AC (AC coupling) and 50 (50 ohm connection) are blue.

Input 2: This is the tapping frequency input from the AFM. Check that AC (AC coupling) and 50 (50 ohm connection) are blue. Set Range to 2.0 Vpk or click the *A* button to autoscale.

Demodulators, Filters, Readout:

The bottom 2 rows under the Demodulator label control the oscillators for the lock-in. Typically, only Oscillator 1 is used, which should be se to *Signal In 2* (i.e. the Tip Tapping frequency). When selected, the tapping frequency should be displayed and a green light should indicate the lock-in amplifier is *locked.*   If the *locked* button is not lit, check that the AFM head is plugged in, aligned and tuned, and that the *TB Fast Signal* output from the *Nanodrive* Hardware Access is selected.

The first 6 rows under the Demodulator label display the parameters of the 6 demodulated signals with associated harmonics and filters, and are called Demod 1, Demod 2, Demod 3, Demod 4, Demod 5, and Demod 6. For all rows, select *1* under the *Osc* column heading (for Oscillator 1), select *1* under the *Input* column heading (for Input Signal 1), and select *48* under the *dB/Oct* column heading for the strength of the filter.   Typically, the first 4 rows (Demod 1, 2, 3, and 4) are reserved for input into the AFM acquisition and the last 2 rows (Demod 5, 6) for input into the FTIR spectrometer, the primary difference between the two being the effective time constant.

> *AFM acquisition*: For Demod 1, set the *Harm* column to *1*, indicating demodulation at 1st Harmonic. Likewise, Demod 2, is set to *2* for 2nd harmonic, Demod 3 is set to *3* for 3rd harmonic, and Demod 4 is set to *4* for 4th harmonic. For Demod 1-4, the settings for the *Phaseshift* column are generally left at *0*.
>
> The time constant for the AFM acquisition must match the scan rate and sampling of the AFM. Too large of a time constant will blur the images (degrade spatial resolution); too small of a time constant will increase the noise of the image. The Effective Time Constant (TC eff) can be approximated by:
>
> $$TC\ eff = \frac{1}{line\ rate\ (Hz) * number\ of\ samples * 2}$$
>
> Where the number of samples is the number of pixels per line specified in the scan control. For typical scans of ~.3 Hz, a time constant of 3-5 ms is appropriate. Make sure *TC eff* in the last row of the *TC (s)* column is selected, or the numbers will not match.

FTIR input: For Demod 5, set the *Harm* column to *1*, again indicating demodulation at 1ˢᵗ Harmonic. Demod 6 is set to *2* for 2ⁿᵈ harmonic. The time constant *TC (s)* for Demod 5 and Demod 6 are set to *200 μs*. This time constant best matches the slowest scan rate of the spectrometer, without losing higher frequency components. The signal can be improved slightly by appropriate selection of the phase input to the FTIR. You can do this automatically, by pressing the arrow symbol (|←) just after the Phaseshift (deg) input. Typically, this is done during the alignment/tip optimization and will be described later.

Auxiliary I/O:

The Auxiliary I/O tab contains the output parameters of the lock-in amplifier. Adjusting the signal, demodulator, and scale parameters to what is displayed in the Figure 19 is a good starting point. More details are listed below to fine tune the parameters.
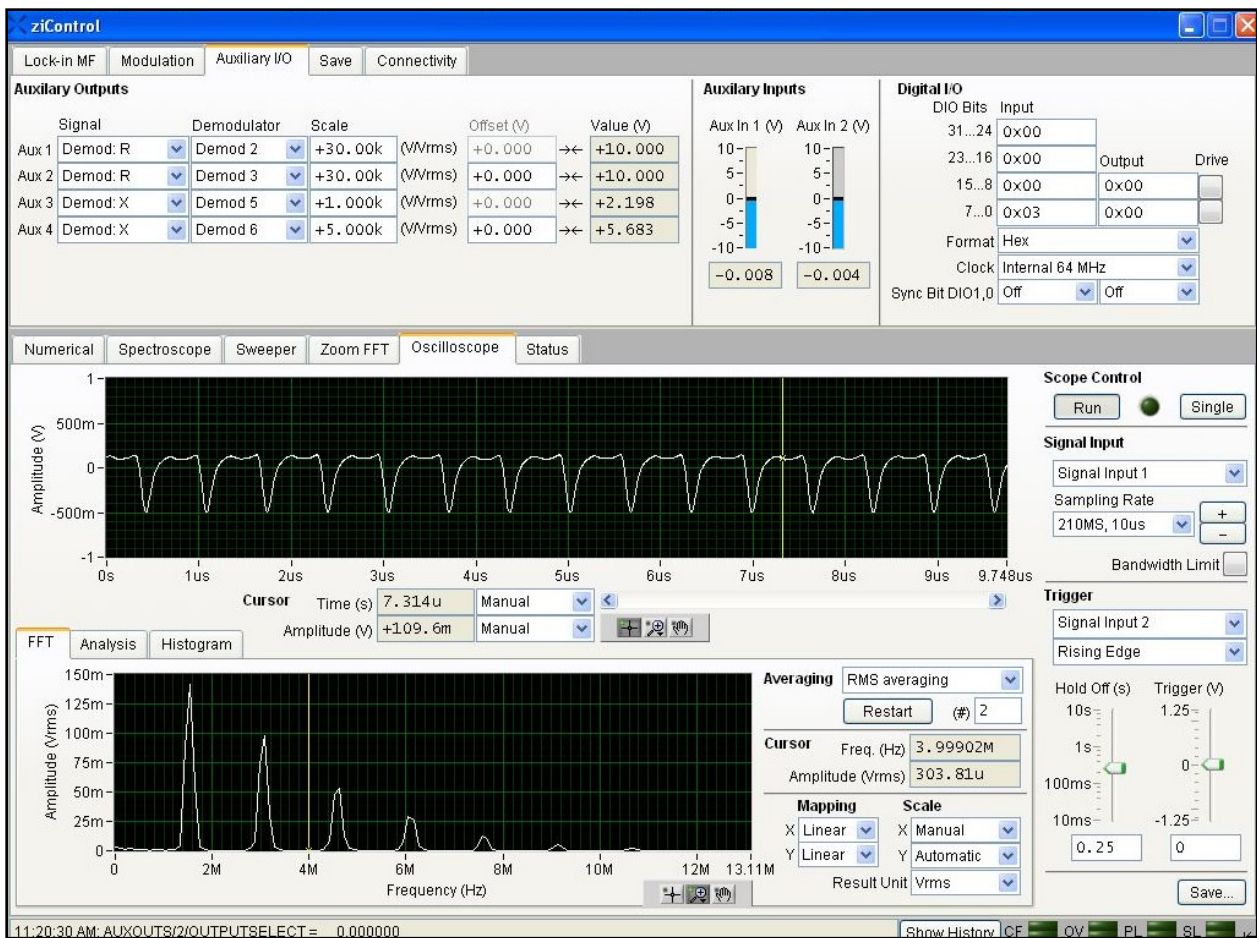

FIGURE 19. AUXILIARY I/O WINDOW. TYPICAL SETTINGS FOR THE AUX TAB OF THE LOCK-IN. THE OSCILLOSCOPE TAB AND FFT DEMONSTRATE BACK-SCATTERED LIGHT IS INCIDENT ON THE DETECTOR. NOTE IN THIS IMAGE, AUX 1 AND AUX 2 ARE SATURATED AT 10 V, INDICATING THE GAIN NEEDS TO BE LOWERED.

Typically, Aux 1 and Aux 2 are reserved as inputs into the AFM control in order to acquire broadband infrared images with an AFM scan and can be set to user preferences.

Aux 1: corresponds to IOC BNC 1 in the AFM software

Signal *Demod:R*, Demodulator *Demod 2* to choose 2nd Harmonic signal (if parameters are chosen as listed above). Depending on the signal level, adjust the *Scale* to change the gain of the lock-in amplifier, which will affect the *Value* column. The *Value* column should be < 10 V to avoid saturation. Typical *Scale* numbers are between 5k and 100k.

Aux 2: corresponds to IOC BNC 2 in the AFM software

Signal *Demod: R*; Demodulator *Demod 3* to choose 3rd Harmonic signal (if parameters are chosen as listed above). Depending on the signal level, adjust the *Scale* to change the gain of the lock-in amplifier, which will affect the *Value* column. The *Value* column should be < 10V to avoid saturation. Typical *Scale* numbers are between 5k and 100k.

~~Aux 3: corresponds to input A of the FTIR~~

~~Signal *Demod: X*; Demodulator *Demod 5* to choose the 1st Harmonic signal (if parameters chosen as listed above). Depending on the signal level, adjust the *Scale* to change the gain of the lock-in amplifier. The interferogram peak-to-peak level should be ~10 V on a gold surface with a gain setting of 1k.~~

~~Aux 4: corresponds to input B of the FTIR~~

~~Signal *Demod: X*; *Demodulator* Demod 6 to choose 2nd Harmonic signal (if parameters chosen as listed above). Depending on the signal level, adjust the *Scale* to change the gain of the lock-in amplifier. The interferogram peak-to-peak level should be ~3 V on a gold surface with a gain setting of 1k.~~

Adjust the phase to ensure the output signal is on X channel. If the autophase (symbol |←) is working well, the numbers shouldn't change too much if the button is clicked again. It may be necessary to adjust the phase manually. The easiest way to check that autophase is working is to select *Demod: Y* for Aux 3 in the Auxiliary I/O tab. The centerburst in Omnic should go to zero, verifying the signal is all on the X channel.

⚠ Omnic has max input of 10 V (keep less than 7.5 max or 15 pk to pk), so adjust the lock-in gain and offset to keep the output below 10 V.

<u>Return to Quick Start Procedure</u>

Omnic Control

The *Omnic* software controls aspects of the spectral acquisition and also controls the FTIR bench. The *Experiment Setup* window is where measurement parameters may be specified. The window can be accessed by navigating to *Select Collect >> Experimental Setup.*

Note: Broadband images are best achieved with the IR source in Omnic turned to "IR", to reduce the noise, as discussed in the *Bench* section below.
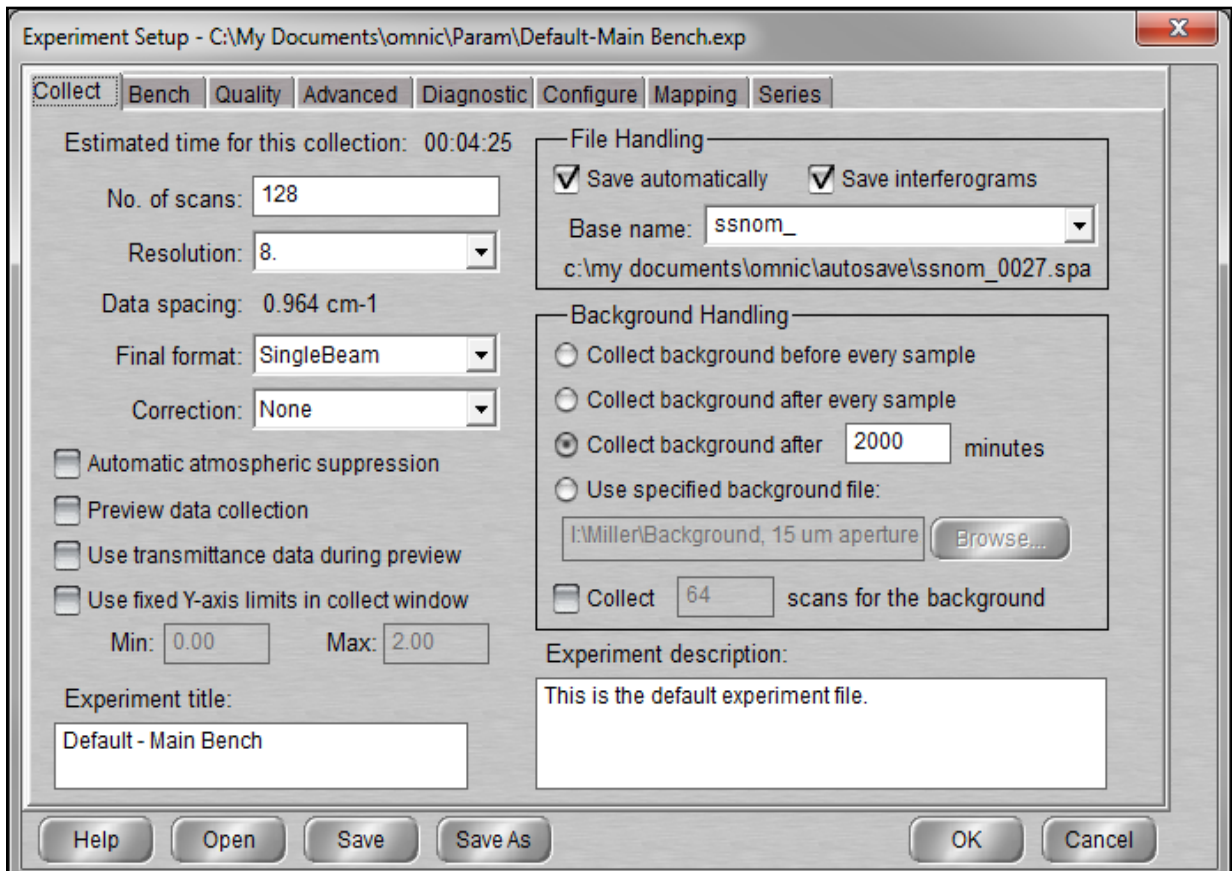

FIGURE 20. COLLECT TAB OF EXPERIMENT SETUP WINDOW. MEASUREMENT PARAMETERS ARE SPECIFIED HERE.

Collect Tab (Figure 20)*:*

> *Number of scans*: Increasing the number of scans will increase the SNR, but will increase the acquisition time.

> *Resolution*: spectral resolution desired. Because higher resolution scans are noisier and take longer, 8 cm^{-1} resolution is typically the highest used for SINS spectra.

Final format: spectra are generally collected in "single beam". The final format can be post-processed at a later time.

Make sure the *Save automatically* and *Save interferograms* box is checked.

Basename: increments with each collect sample and saves in autosave directory. This can be changed with user preference.
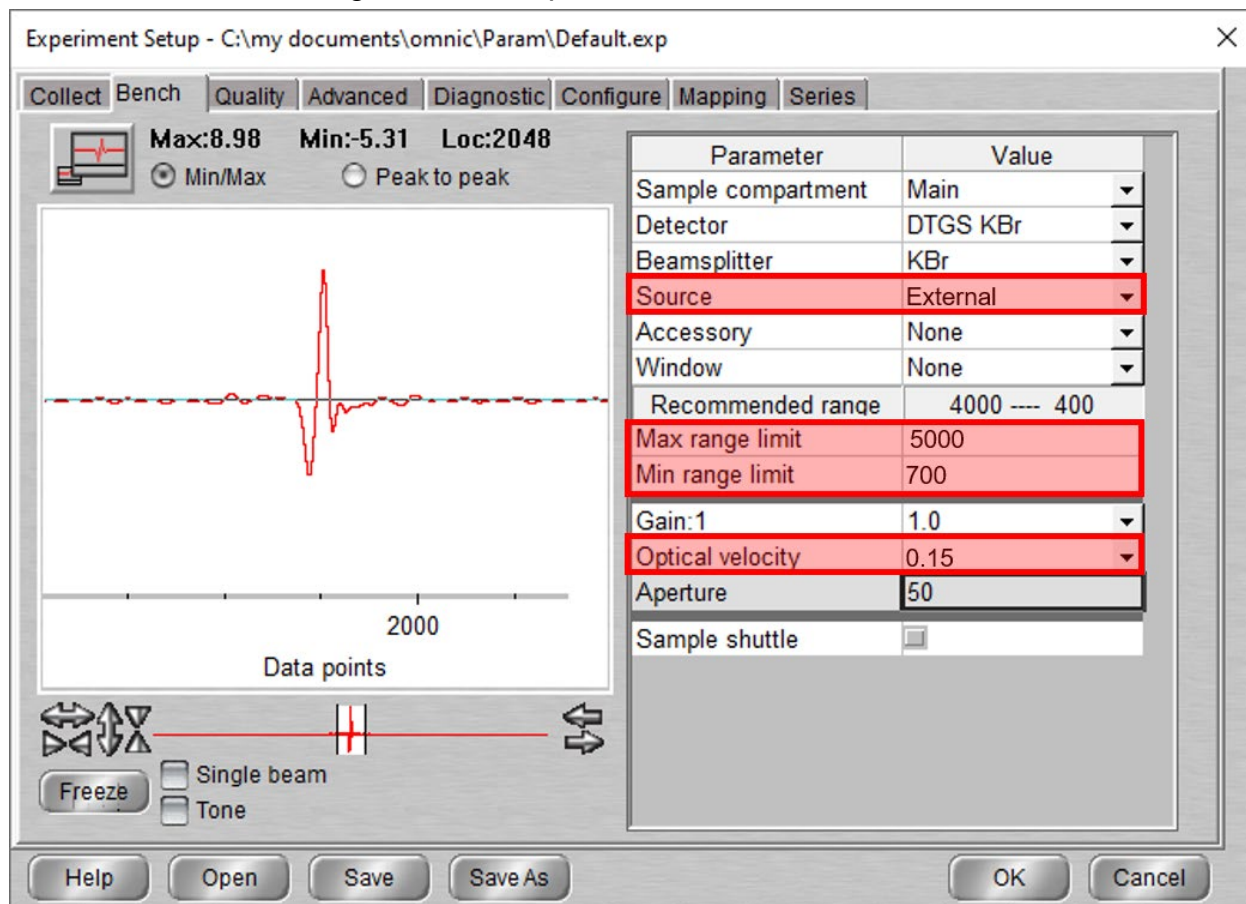

FIGURE 21. BENCH TAB. RELEVANT PARAMETERS ARE HIGHLIGHTED IN RED. A TYPICAL CENTERBURST IS SHOWN IN THE LEFT PART OF THE WINDOW.

Bench Tab (Figure 21)**:**

Source: Set to <u>*External*</u> to select the synchrotron beam for spectral acquisition.
Set to <u>*IR*</u> to block the reference arm path for broadband imaging.

Scan Range: The Kolmar detector has a low-frequency cutoff at 700 cm^{-1} and a high-frequency decay around ~ 5000 cm^{-1}.

Scan speed: Typically set to 0.15 cm/s when the *ziControl* time constant is 200 µs. The speed can be increased with appropriate decrease in time constant.

The centerburst should be visible in this tab. If no signal is present, verify light is available, not blocked, and see the Troubleshooting section "No Interferogram".

<u>Return to Quick Start Procedure</u>

Collecting Spectra

There are two options for collecting spectra: (1) single channel (2) and dual channel:

Collecting single channel spectra: Recommended if the signal is strong and only one data channel desired.

1. Check that the correct signal is selected for Aux 3 in the *ziControl* software. Typically, this will be the 2nd harmonic signal:

    Aux 3: Signal Demod: X; Demod 6

2. Check parameters for appropriate resolution and acquisition time.

3. If Single Beam is selected as Final Format in Experimental Setup, proceed to (4). If %reflectance is selected, then:

    Go to an appropriate place on the sample to collect a background with the Nanodrive AFM software
    i. Select Collect >> Background
    ii. Collect Sample

4. Go to an appropriate place on the sample with Nanodrive AFM software:
    i. Select Collect >> Collect Sample

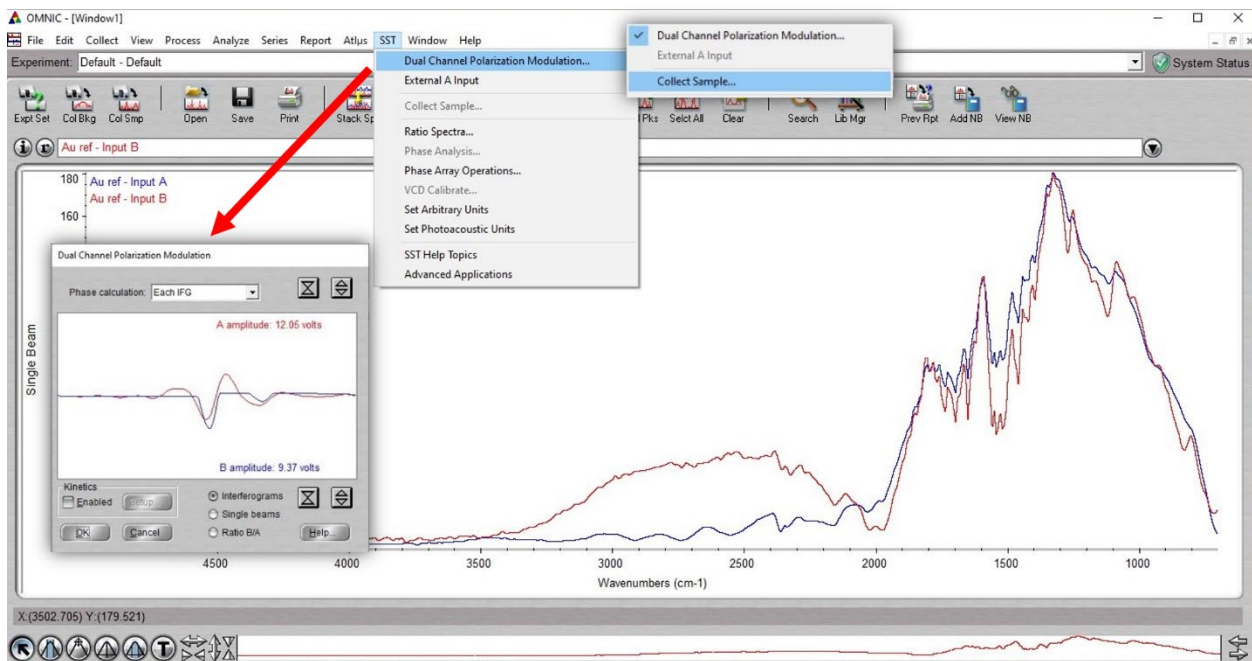   You will be prompted for a title, which can be used to document the sample.

FIGURE 22. DUAL CHANNEL SST DATA COLLECTION. NOTE THE DUAL CHANNEL POLARIZATION MODULATION SHOWN IN THIS IMAGE IS SATURATING THE DETECTOR (BLUE/B INPUT) AND THE GAIN NEEDS TO BE ADJUSTED. AFTER CLICKING "OK", PROCEED TO SST >> COLLECT SAMPLE.

Collecting dual-channel spectra: collects multiple harmonics simultaneously.

1. Check that the correct signal is selected for Aux 3 and Aux 4 in the *ziControl* software. Typically, this will be the 1st harmonic signal for Aux 3 and 2nd harmonic signal for Aux 4:

   > Aux 3: Signal Demod: X; Demod 5

   > Aux 4: Signal Demod:X; Demod 6

2. Check the parameters for the appropriate resolution and acquisition time and select *Single Beam* as *Final Format*.
3. Select *SST >> Dual Channel Polarization Modulation*
4. Collect Sample
   a) Go to an appropriate place on the sample with *Nanodrive* AFM software:
   b) Select *SST >> Collect Sample*. You will be prompted for a title, which can be used to document the sample. Note: ziControl Aux 3 corresponds to Channel A in Omnic and ziControl Aux 4 corresponds to Channel B. Only the interferograms will be displayed during the collection.

Tip Change

Removing the tip:

Retract the tip using the *Withdraw* button (circled in red below) in the *Engage* window.

To minimize re-alignment time, align red crosshairs in the *Navigator* window with AFM tip using objective controls and <u>*take a picture of the tip location*</u>.

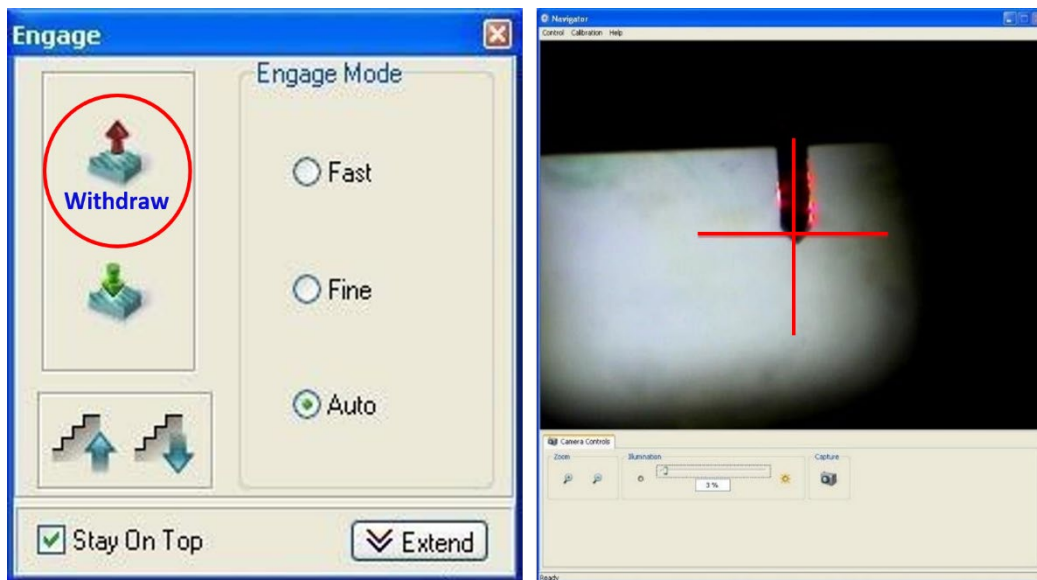> *This will significantly aid in bringing the new tip to the location of the IR beam*!

FIGURE 23. ENGAGE AND NAVIGATOR WINDOWS. WITHDRAW THE TIP AND THEN ALIGN THE OPTICAL MICROSCOPE TO THE OLD TIP POSITION BEFORE REMOVING AFM HEAD.

FIGURE 24. TIP CHANGE BOX. THIS BOX CONTAINS THE TOOLS NECESSARY FOR A TIP CHANGE. PLEASE RETURN ALL TOOLS TO THIS BOX WHEN TIP CHANGE IS COMPLETE.

Remove the AFM head from the microscope base, being careful not to bump the objective.

Open up the *Tip Change* box, which contains the tools for removing and replacing tips. With the small screwdriver in the *Tip Change* box, lever the tip cartridge from AFM head.
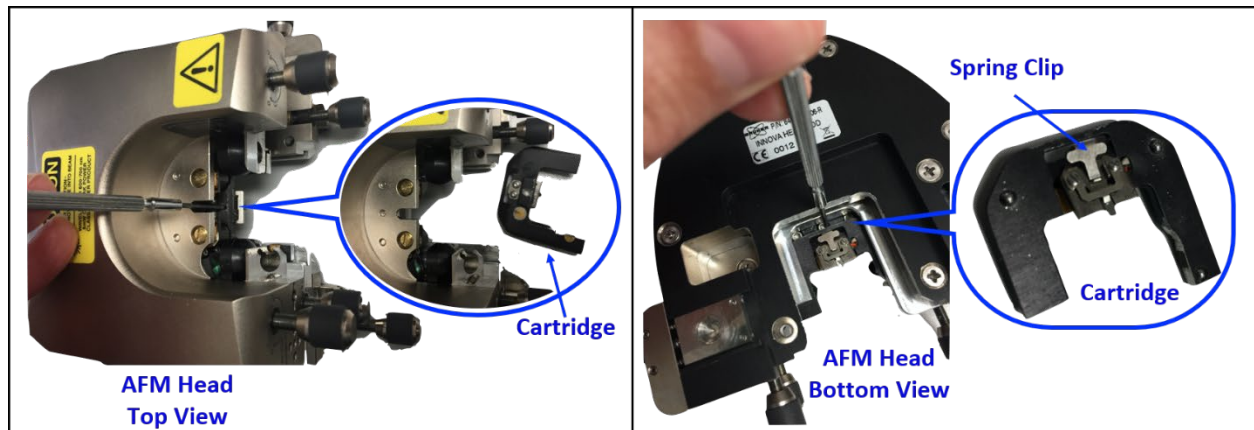

FIGURE 25. EJECT CARTRIGE FROM AFM HEAD. LEFT: TOP VIEW AND RIGHT: BOTTOM VIEW OF TIP CARTRIDGE BEING LEVERED FROM THE AFM HEAD WITH THE SMALL SCREWDRIVER.

Use the special lever tool to remove metal chip carrier from the cartridge. The lever tool is used to lift the spring clip which holds the chip carrier in place. While the spring clip is lifted, remove the chip carrier. The suggested finger positioning is to place the thumb and index finger of left hand on either side of the tip cartridge and use the little finger of the left hand to press on the lever tool. While pressing the lever tool, use the right hand to remove the chip carrier.
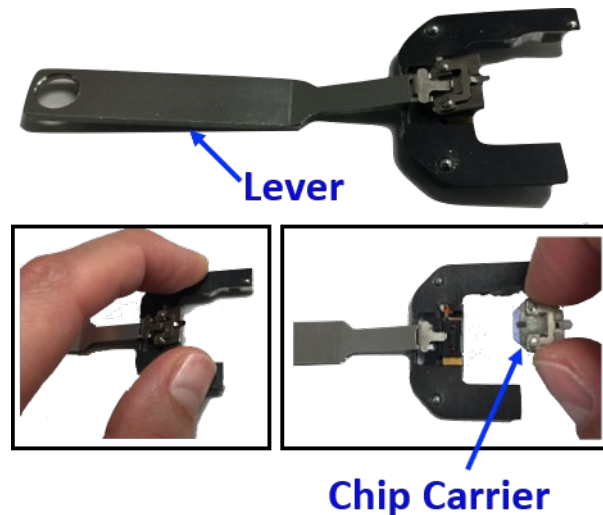

FIGURE 26. LEVER, CARTRIDGE, AND CHIP CARRIER. THE TOP IMAGE SHOWS THE LEVER TOOL INSERTED UNDER THE SPRING CLIP TO ENABLE CHIP CARRIER REMOVAL. THE LOWER LEFT IMAGE SHOWS THE SUGGESTED FINGER POSITIONING FOR USING THE TOOL. THE CHIP CARRIER AFTER REMOVAL FROM THE CARTRIDGE SPRING CLIP CAN BE SEEN IN THE LOWER RIGHT IMAGE.

Pre-mounted tips and cartridges are available in the clear box labeled *New Tips* - these should be used first. Reverse the steps above to insert a new chip carrier into the cartridge by lifting the spring clip with the lever, and then slide the cartridge back into the AFM head. Use extreme caution when handling the mounted tips. Do not to touch the chip/cantilever with either fingers or tweezers.

⚠️ If there are no pre-mounted tips available, contact the Beamline staff for assistance. If the staff are not available, advanced Users can proceed with the tip change procedure detailed below.

Place the metal chip carrier on the black mounting block. The block has small pins which will lift the clamp holding the chip in place. Make sure the chip carrier is seated on the pins. Take a moment to look closely at the position of the old chip relative to the carrier – specifically notice if the chip is at an angle (gray line down center of chip), closer to one side (white lines and arrows in the image below), or extended/retracted relative to the front edge of the carrier (red dashed line in image below).
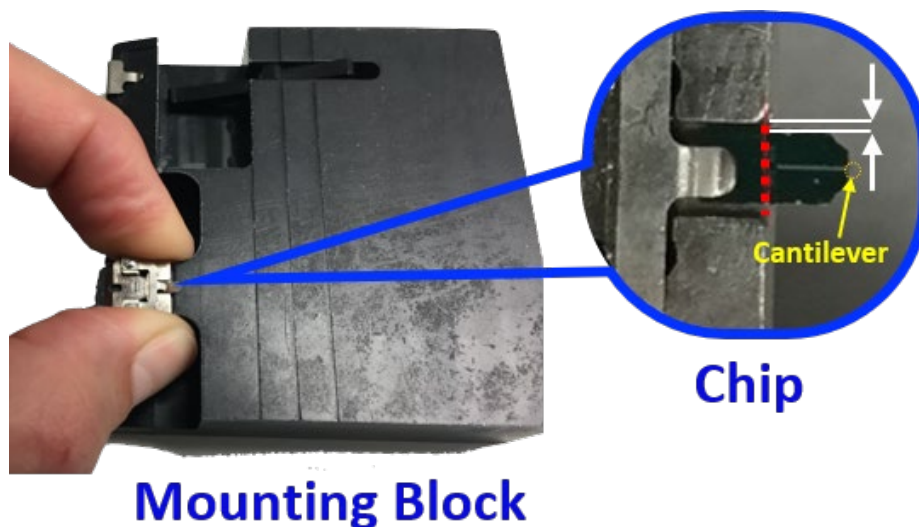

FIGURE 27. MOUNTING BLOCK AND CHIP. IMAGE OF THE CHIP CARRIER SEATED ON THE MOUNTING BLOCK. THE INSET SHOWS THE CHIP MOUNTED IN THE CHIP CARRIER. NOTICE THE RELATIVE POSITION OF THE OLD CHIP IN THE CARRIER AND TRY TO POSITION THE NEW CHIP IN THE SAME WAY.

Press down on the sides of the chip carrier while using tweezers to pull out the tip. Place the old tip in the *Used Tips* box and carefully remove a new tip, grasping the chip firmly on the sides with tweezers and wiggling slightly to release it from the gel pack. Set it down carefully on the mounting block, if needed, while preparing to insert the new chip into the carrier. Press down on the sides of the chip carrier to lift the clamp and slowly insert the new chip.

Note: the cantilever chips are mounted with the tip pointing up in the gel packs: do not flip the chip upside-down or the tip will be destroyed.

Align the horizontal line on the chip with edge of the carrier and reproduce the lateral positioning for consistency.

Place the chip carrier into the AFM cartridge: using the lever tool to lift the spring clip while inserting the carrier with tweezers. The chip carrier should be seated on the ruby nodules.
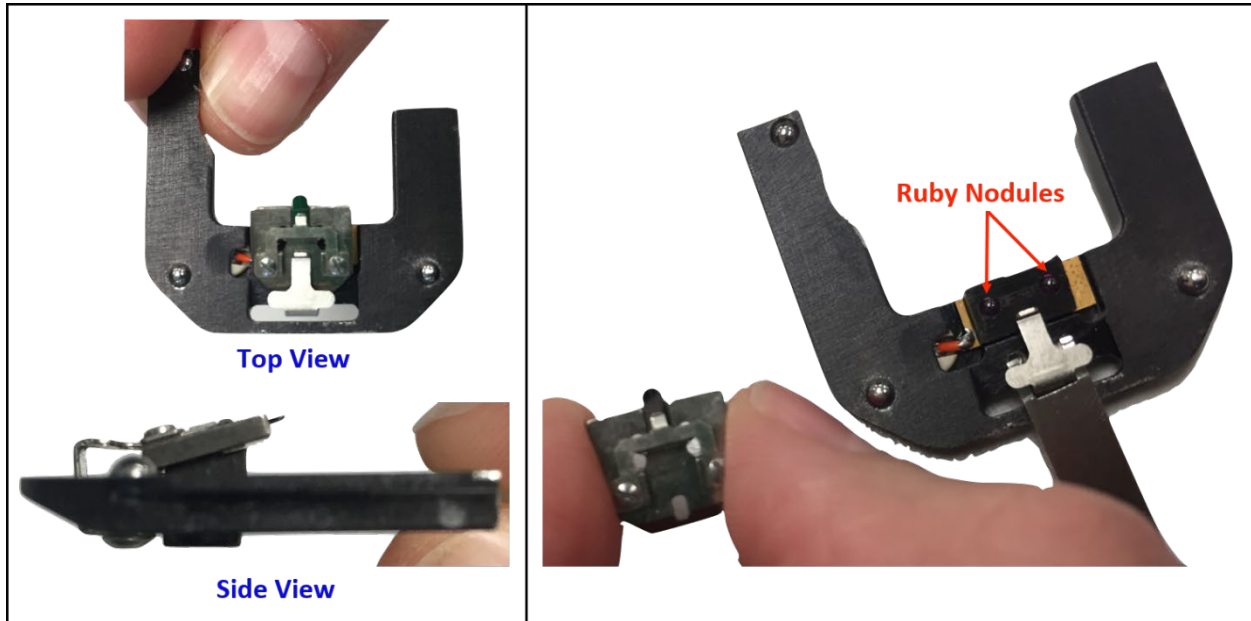

FIGURE 28. INSERTING THE CHIP CARRIER INTO THE CARTRIDGE. LEFT PANNEL: TOP AND SIDE VIEWS OF THE MOUNTED CHIP CARRIER. THE RIGHT PANEL SHOWS THE RUBY NODULES PLACEMENT PINS. THE CARTRIGE SHOULD BE FIRMLY SEATED ON THESE NODULES.

After verifying the carrier is firmly in place, slide the cartridge back into the AFM head (upper holder track).

Verify the sample stage is lowered, then replace AFM head on microscope being careful not to bump the objective. Reconnect the electrical cable for the AFM head. Align the cantilever to red crosshairs using the *Tip Positioners* on the AFM head. Only very small adjustments should be made with the *Tip Positioners*; if the tip is not near the center of the crosshairs, remove the head and adjust the placement of the cantilever chip in the chip carrier. Proceed to the laser alignment and autotune instructions.

Optical Alignment

Parabolic Mirror Alignment:

This is the primary adjustment for light onto the tip. Each time the AFM head is removed, the parabolic mirror will likely need to be adjusted slightly for maximum intensity. Typically, this just involves small tweaks (quarter turns of the fine adjustment knobs) of the horizontal and vertical motions. When a tip is changed, more severe motions are likely, particularly if the tip is in a different focal plane. A scattered light signal can be used as a guide to the eye.

⚠️ *If it appears that more than quarter turns of the fine adjustment knobs are needed, contact Hans or Stephanie.*

Detector Alignment: (*Advanced Users Only*)

Small tweaks to the detector position may improve signal.

Nominal micrometer positions (Kolmar detector):

      X (horizontal plane, parallel to long axis of table): 3.93

      Y (horizontal plane, perpendicular to long axis of table): 6.94

      Z (vertical plane): 12.00

Interferometer Alignment: (*Advanced Users Only*)

This arm typically does not need to be adjusted during a normal user run; however, if a complete alignment is being performed, the final mirror before the bench can be used to fine tune the interferogram signal. Small adjustments can dramatically change the signal. Typically, the best signal corresponds to the maximum intensity on the detector. This should primarily be done by the Beamline Scientists.

Programming Measurement Positions

Line or Area Scan:
1) ~~Move coax cable from the Aux2 output on the Zurich lock-in amplifier to the AO 0 output on the National Instruments BNC 2110, located on the floor under the optical table.~~
2) In the *Nanodrive* software, select *Realtime Control >> Synchronizer Control*.
3) Make sure that the Enable button is checked and the Handshake Input button is checked. Press *Apply*. This will cause the laser alignment window to turn gray.
4) Open the *Probe Positioning* Dialog window (icon with arrow target or *Realtime Control >> Probe Positioning*).
5) In the *Probe Positioning* Dialog window, select *Distance* (instead of Volt), and change the wait time to *-1*.
6) In the Scanning window, draw a line (or mark points or define an area) on the part of your sample with the desired number of points.
7) In the *Probe Positioning* Dialog Window, select *Save* to save the positions to a text file for later retrieval. Also, take a screenshot (*Alt + Print Screen*) of the scanning window to show the area of the scan.
8) In *Omnic*, change the base name in *Collect >> Experimental Setup >> Collect* to indicate your linescan.

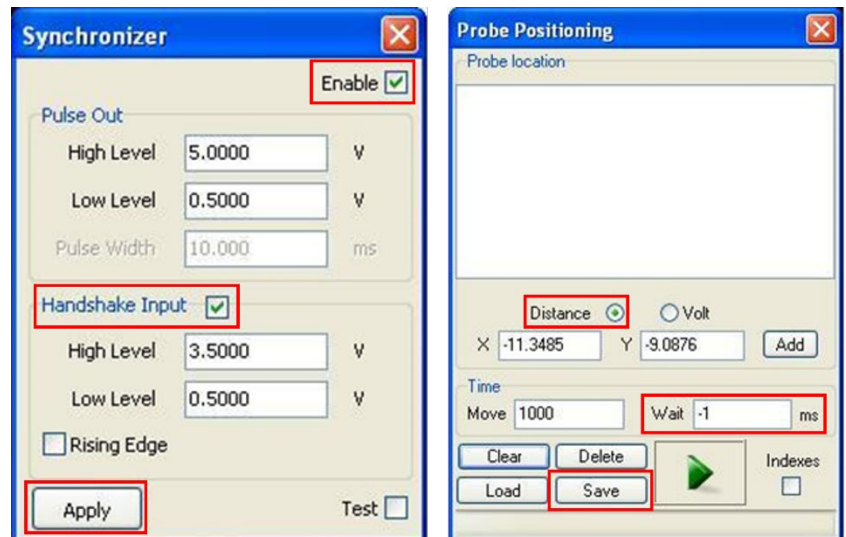**FIGURE 29. NATIONAL INSTRUMENTS BNC 2110.**

FIGURE 30. SYNCHRONIZER CONTROL WINDOW AND POSITIONING DIALOG WINDOW. THE SYNCHRONIZER WINDOW IS SHOWN ON THE LEFT AND THE POSITIONING DIALOG ON THE RIGHT. MAKE SURE TO CHANGE THE WAIT TIME TO (-1) IN THE PROBE POSITIONING TO ENABLE THE FTIR TO TRIGGER THE PROBE MOVEMENT.

9) Open *Labview* and select "*Omnic Step_dual_input.vi*" (for small data sets) or "*Omnic Step_dual_input_clear.vi*" (for large data sets) to record two channels of data.

10) In the *Labview* program .vi, select the number of steps to match the number of points chosen in the *Nanodrive Probe Positioning* Dialog window.

11) Press the green arrow in the *Nanodrive Probe Positioning* Dialog window.
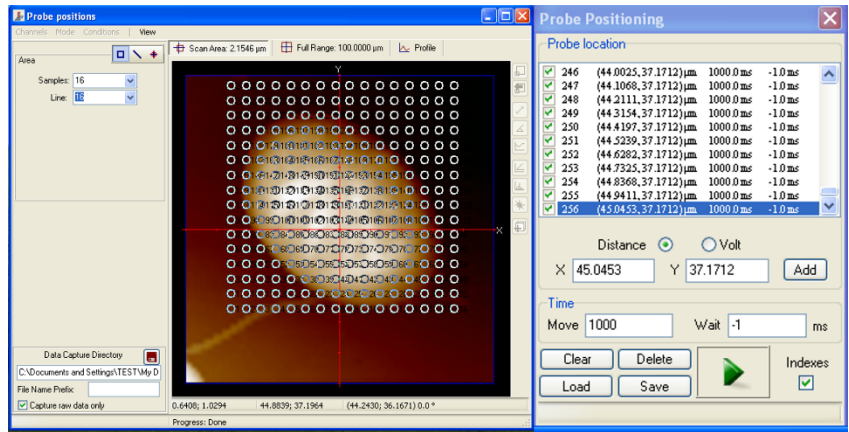
12) Press the start arrow for the *Labview* program.

FIGURE 31. EXAMPLE OF PROBE POSITIONING LOCATION DETAILS FOR A LARGE MAP. USE *"...INPUT_CLEAR.VI*" FOR LARGE DATA SETS.
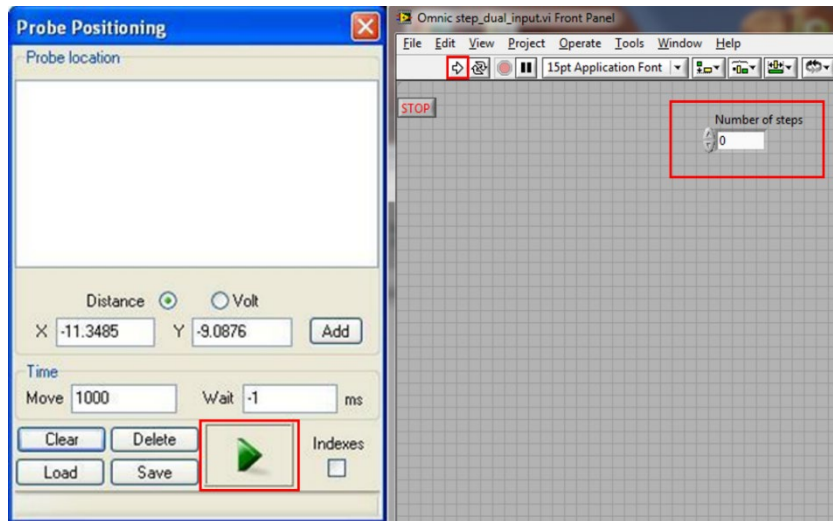

FIGURE 32. POSITIONING DIALOG AND LABVIEW DUAL CHANNEL DATA COLLECTION WINDOW. PRESS THE GREEN PLAY BUTTON ON THE PROBE POSITIONING WINDOW BEFORE PRESSING THE START ARROW IN THE LABVIEW VI. *NOTE: THE NUMBER OF PROBE LOCATIONS TO BE MEASURED SHOULD MATCH THE NUMBER OF STEPS IN THE VI.*

Troubleshooting

Lock-in indicator in ZI Control software is dark (green when active):

Check that *Nanodrive* software has been properly configured using *Open Hardware Access*. TB fast signal should be selected for the output BNC as indicated in Figure 33.
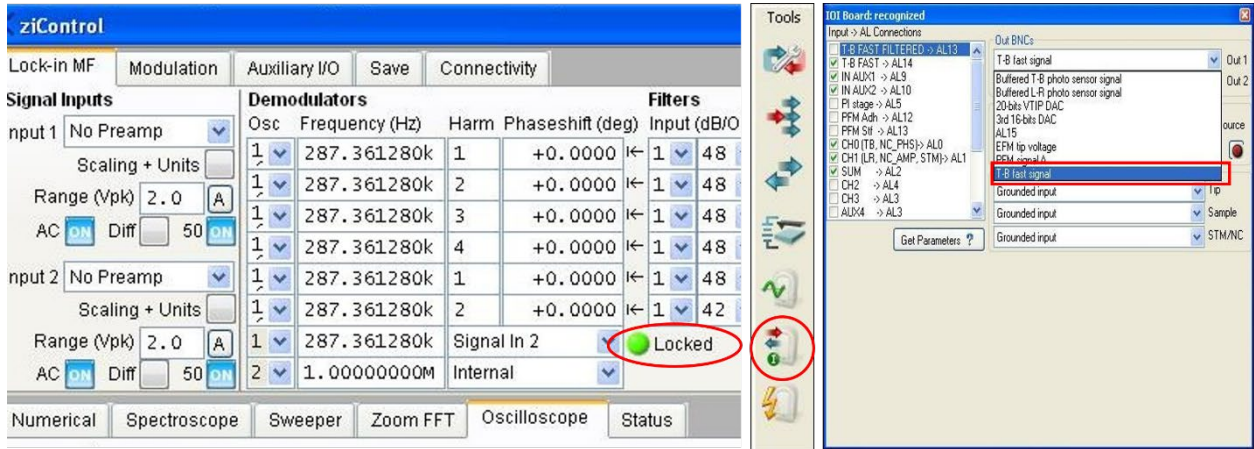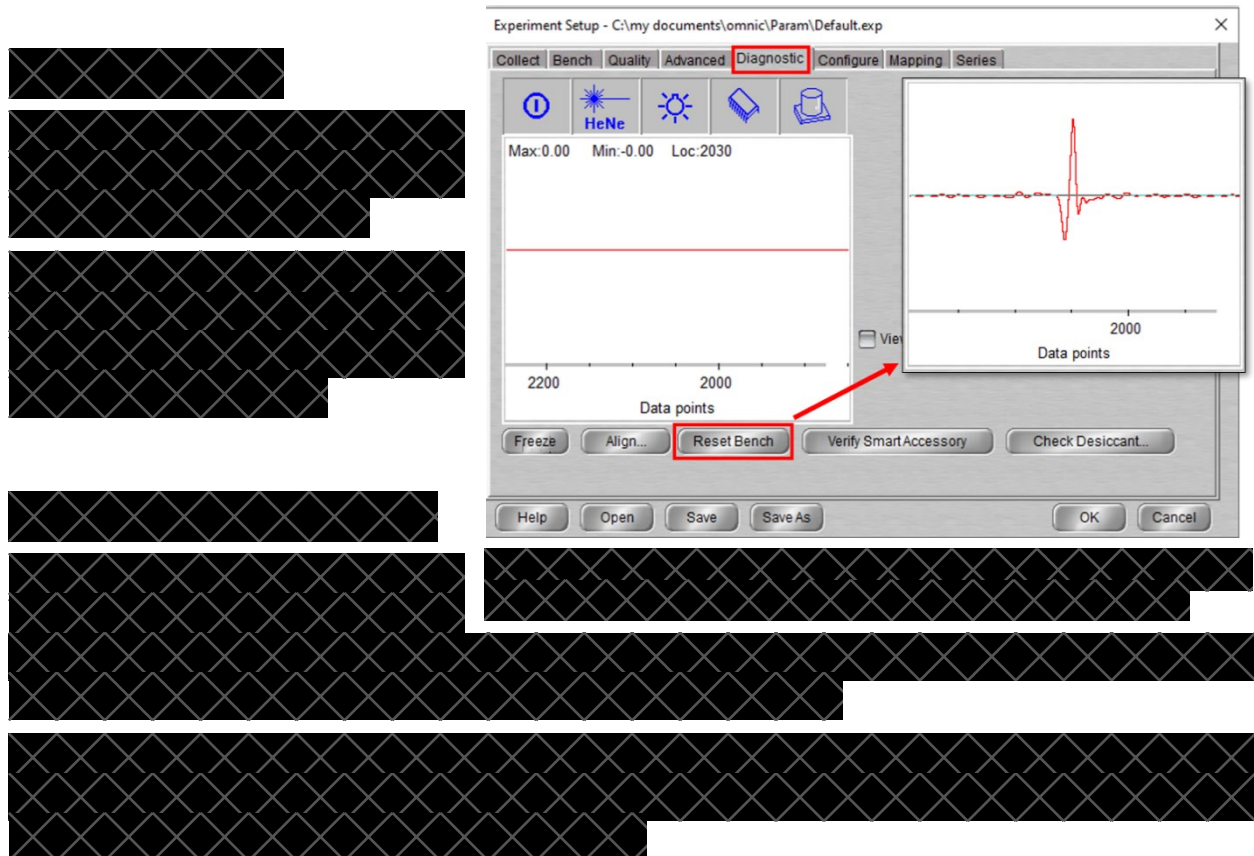

FIGURE 33. ZICONTROL. AT LEFT, SHOWING ACTIVE LOCKING. IF NOT ACTIVELY LOCKED, OPEN HARDWARE ACCESS IN NANODRIVE AND THE IOI BOARD (MIDDLE PANEL) AND MAKE SURE THE T-B FAST SIGNAL IS SELECTED FOR THE OUTPUT BNC.

AFM Navigator has blue screen:

Wiggle the BNC cable that is attached to the AFM Video output.

AFM sample stage tilts forward:

The sample stage stepper motors are withdrawn in the vertical (z) direction too far and need to be raised. With the head removed, open the motor stage dialogue and click "Stage Reset". After resetting (moving to the stage's lowest point), push the down arrow in the top of the dialogue until the stage levels out. Note: the motor was reversed when the AFM was converted to stage approach from tip approach; i.e. down is up.

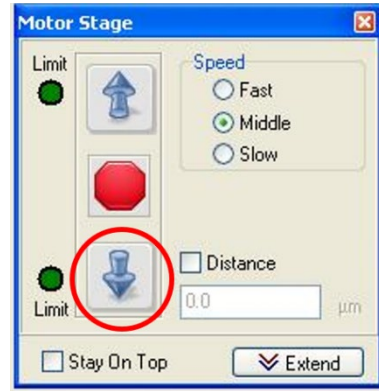You may need to change the sample mount so that it is thinner to prevent this from occuring.
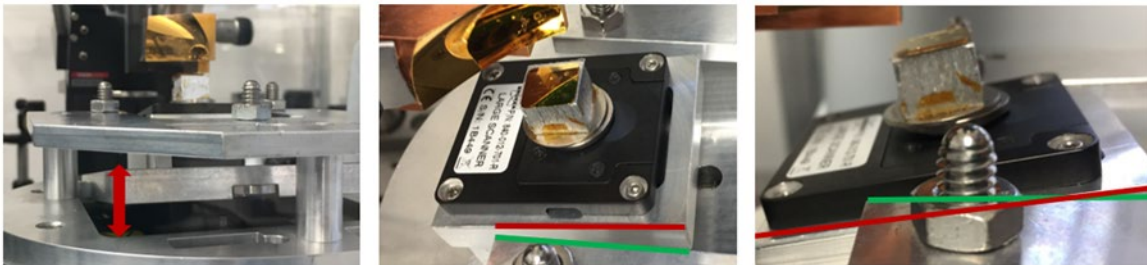

FIGURE 35. AFM MOTOR POSITION DIALOGUE.

Figure 36. TILTED AFM STAGE. FREQUENTLY OCCURS WHEN THE SAMPLE IS TOO THICK FOR THE CHOSEN SPACER BLOCK.

Trouble finding alignment laser after tip change:

Looking down from over the AFM head, the red deflection laser should be visible in some region of the cantilever/tip holder. Walk the beam by adjusting the laser positioning actuators until the laser beam is most of the way down the cantilever. Maximize the signal, and then adjust the PSD.

Alignment laser walks or jumps:

If the alignment laser spot drifts on the position sensitive detector (PSD) significantly over the course of a few minutes (i.e. across the width of the detector) *OR* if the laser location jumps on the sample surface during alignment of the laser via the AFM head laser vertical/horizontal screws, contact Hans or Stephanie as the AFM head may need immediate repairs.

Sample drift:

If you experience significant drift, minimize the amount of time the nitrogen purge lid is removed and consider adhering your sample to the aluminum block with a different method. Carbon tape and copper tape perform better in terms of drift than double-stick tape. Fingernail polish, silver paste, and glues work best, but are harder to remove from your sample. The longer the sample thermalizes in the purge box with the lid closed, the less likely drift will impact your measurements. If your sample is not centered on the magnet, you may also get increased drift.

Tip not making contact:

If the tip isn't making good contact (scans don't seem to follow the topography of the sample well), disengage to lower the sample stage. Then, verify the AFM deflection laser position on the detector is within the appropriate bounds. Re-tune the cantilever using the full frequency sweep (0 - 1000 kHz). Remember to adjust the setpoint to ~ 3.7 V and engage the tip. If the tip still doesn't make good contact after the above steps, try lowering the setpoint or adjusting the drive voltage by small increments (0.1 V) until the topography forward and backward trace appear to follow the sample surface.

Sinusoidal oscillations in spectra:

If unphysical oscillations are present in the spectra, it is likely an issue with the feedback for the AFM. Try adjusting the gain settings until the oscillations disappear from the spectra.