SOP for Bruker FT-IR Spectrometer & Microscope

Note: This document is frequently updated; if you feel that information should be added, please indicate that to the facility manager (Currently Philip Carubia; pmc228@cornell.edu; Office, Bard B56; Phone, 845-551-6501).

- **Reservations and enabling on coral**
  - The Bruker Hyperion FT-IR Spectrometer and Microscope is on the CCMR Coral equipment Reservation and enabling system. You need to have a CCMR user account so that you can reserve and enable the equipment on Coral. More Information on getting an account and using coral is at: http://www.ccmr.cornell.edu/facilities/coral.html
- **Log book**
  - Record the date, your name and hours of usage. Comment on problems.

About the FT IR

- **Detectors**
  - There are two detectors associated with the FT-IR an MCT and a DTGS. The MCT Detector is approximately 50 times more sensitive than the DTGS and it requires liquid nitrogen for operation. The MCT Detector is located in the Microscope and the DTGS detector is located in the spectrometer. The MCT detector is only available when using the microscope and the DTGS detector is available only when using the spectrometer. The MCT detector requires Liquid nitrogen for cooling; please see section on turning on equipment for filling procedure. Note: Liquid nitrogen is extremely cold ≈-196°C at atmospheric pressure. When filling ensure that you are wearing safety glasses, cryogenic gloves and closed toe shoes.
- **Microscope**
  - **4x:** This is a coarse objective. It is useful for coarse focusing and observing the specimen with a wide field of view. This objective has quartz lenses; Quartz absorbs light in the Mid Infrared region so this objective is not useful for taking spectra.
  - **15x:** This is the standard objective for the microscope. With this objective you have the ability to view your sample with visible light and take spectral measurements in reflectance and transmittance mode.
  - **ATR (attenuated total reflectance):** This objective uses a germanium crystal to reflect light off of the interface between the specimen and crystal. Light is reflected several times prior to collecting the spectrum. This objective allows

easy measurement of a large range of specimens. The nature of this measurement technique requires that the crystal be in contact with your specimen. Crystal tip diameter is ≈100µm. The crystal refractive index is 4. For good measurements sample refractive index must be less than that of the crystal. Two hours of equipment operation time and a second training session is required to use this objective.

- **GIR (Grazing Incidence Reflection):** The GIR objective is optimized for measurements of very thin layers on highly reflecting substrates. This objective projects light onto the sample at a high angle "grazing" the surface. This lens maintains the orientation of polarized light; with P-polarized light leading to enhanced absorption and S-polarized light showing little or no absorption. Because of this, the exclusion of S-polarized light can enhance the sensitivity. Two hours of equipment operation time and a second training session is required to use this objective.

Microscope parts

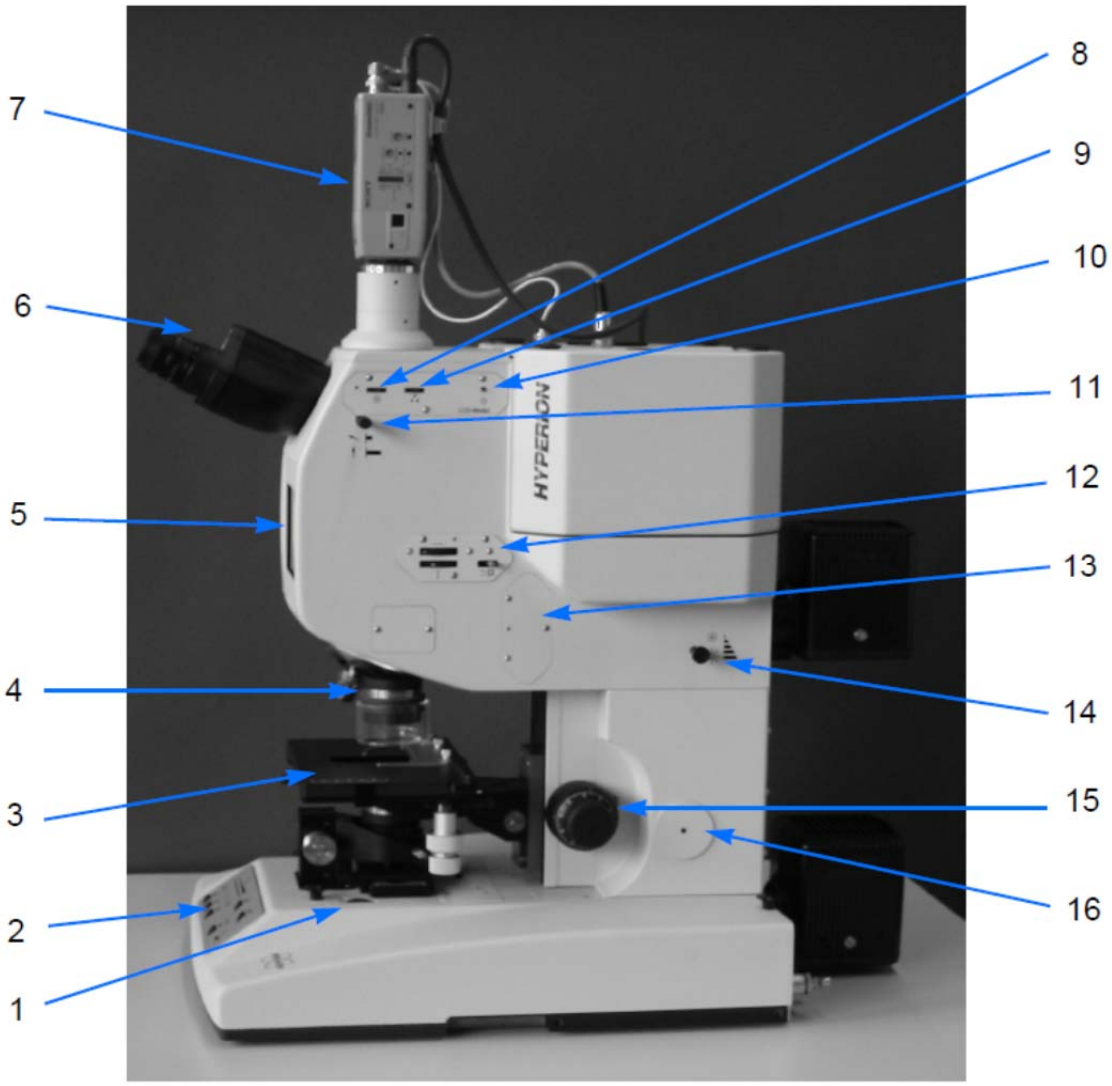

Figure 2: HYPERION - Right Side View

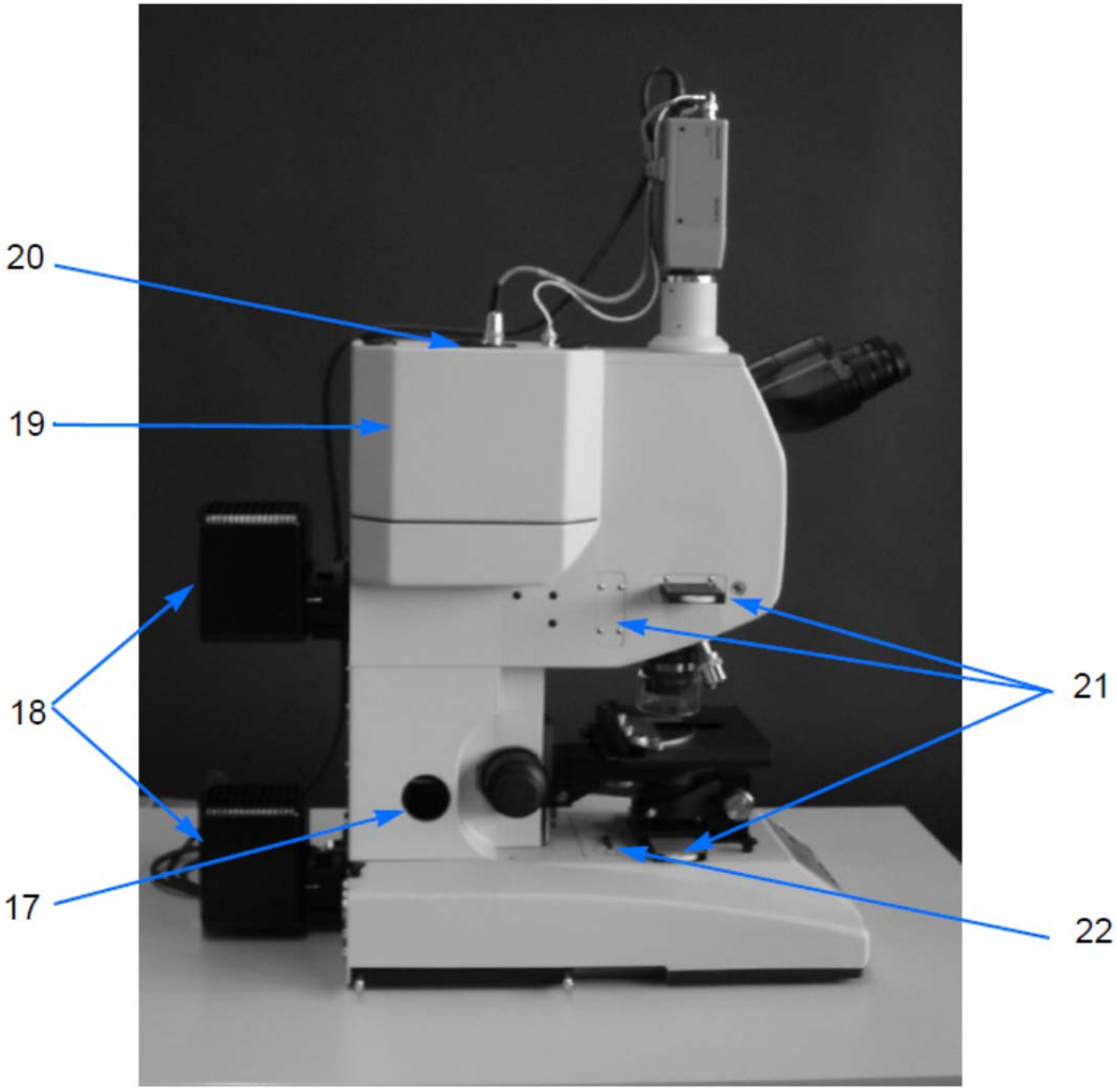

Figure 3: HYPERION - Left Side View

Number	Component
1	Visible light intensity control
2	Control panel
3	Manual sample stage (standard)
4	Revolving turret with objective
5	LCD monitor (optional)
6	Binocular eyepiece
7	Video camera
8	Illumination control of LCD monitor (optional)
9	Color control of LCD monitor (optional)
10	On/Off switch of LCD monitor (optional)
11	Visible light routing control (ocular/video camera)

Number	Component
12	Aperture position (in this case: transparent knife edge aperture)
13	Optional aperture installation site
14	Köhler aperture control
15	Focussing controls
16	External accessory port
17	Spectrometer port
18	Visible light sources
19	Detector compartment
20	Detector filling port (for liquid nitrogen)
21	Polarizer installation sites
22	Alignment aperture

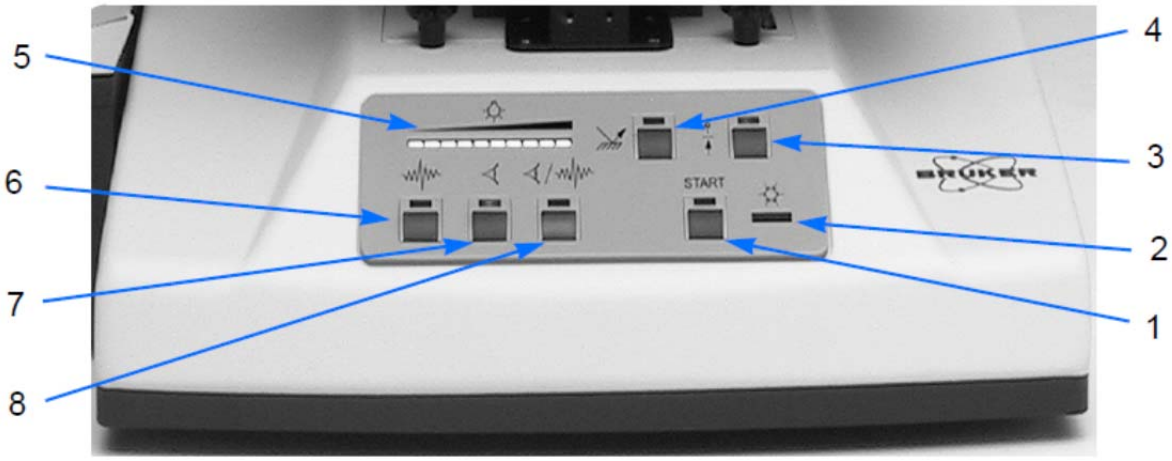

Figure 4: Control Panel

Number	Component and Description
1	**Start button:** starts a measurement directly from the microscope. Pressing this button sends a signal to the computer and the OPUS software acquires a spectrum using the current measurement parameters. **Note: Before pressing this button ensure that the OPUS software is started and set up correctly. Otherwise no measurement can be performed.**
2	**Detector temperature warning indicator:** lights up when the MCT detector temperature is not within its optimum range. The reason for it is either a low liquid nitrogen level in the detector dewar or a continuous cooling down of the detector after a recent dewar refill.
3	**Transmittance mode button:** collects data in the transmittance mode. Pressing this button automatically turns off the reflectance mode button (4).
4	**Reflectance mode button:** collects data in the reflectance mode. Pressing this button automatically turns off the transmittance mode button (3).
5	**Visible light intensity indicator:** indicates the current illumination in relation to the maximum illumination on a bar graph display. The visible light intensity is controlled by a thumbwheel. (See figure 5.)
6	**IR button:** sets the microscope for IR measurement. Ensure that this button is pressed before starting an IR data acquisition. Pressing this button automatically turns off the VIS button (7) and the VIS/IR button (8).
7	**VIS button:** sets the microscope for viewing the sample through the eyepiece or on the LCD display. Pressing this button automatically turns off the IR button (6) and the VIS/IR button (8).
8	**VIS/IR button:** enables simultaneous viewing of the sample and IR measurement. (Transmittance Mode - A 689-T) (Reflectance Mode - A 689-R)

Getting Started

- **Equipment to be left on at all times**
  - By default the power to the spectrometer and the microscope should remain on at all times. The only time that the power should be cycled is if the unit is having trouble communicating with the computer. This will manifest itself as with a lack of ability to switch modes between microscope and spectrometer or transmission or reflection.
    - Spectrometer power switch is located on the rear left side between the two heat sinks.
    - Microscope power is located on the rear right side.
  - The MCT detector requires cooling using liquid nitrogen. To fill the detector you must first fill the 1 L Dewar from the 50 L Dewar. There is an LN2 fill log, please use this log when you fill so future users can judge the level of LN2 in the detector. When fully charged with LN2 the detector should stay cool for 6 to 8 hours.
    - Enter ID and date and time in the LN2 fill log.
    - To fill the 1 L Dewar: Adorn required safety gear; safety glasses, cryogenic gloves, and closed toe shoes. Place the 1 L Dewar on the floor next to the 50 L Dewar. Gently place the filling tube in the small Dewar (It is glass and is easily breakable) and slowly open the Liquid valve. It will take some time for the N2 gas to evacuate the hose and before LN2 will begin flowing. Be cautious to not open the liquid valve to much as the bottom of the small Dewar is curved and it will shoot LN2 back at you. Fill the Dewar ~¼ full.
    - To fill the detector. Remove the plug on the top of the microscope exposing the detector fill tube. Insert the liquid nitrogen funnel into the tube. Pour from the 1 L Dewar into the funnel. A room temperature detector will take approximately 4 funnels worth of LN2. Try not to overfill the detector; if it is overfilled, LN2 will spill across the top of the microscope, Step back and allow excess to run off without spilling on you. If the detector isn't warm you will have to gauge how much LN2 to add from the fill log. Item number 2 in figure 4 is the detector temperature warning light. This light will be red if the detector is hot and off if the detector is cool.
- **Log on to computer**
  - Log on using the FTIR User Account. The Password is Ftirisawesome
  - Open CORAL, sign on and activate the FTIR for your session.

- **Start OPUS**
  - Double click the OPUS 6.5 Icon
  - Log in screen will appear enter the following fields
    - Find your user ID (it will be your Cornell ID)
    - The default password is OPUS
    - Press login button; press OK on the about OPUS Pop up window.
    - You can change your password from the default once in OPUS.
  - You have been assigned a workspace file. This file has the extension .ows. This file is specific to your user account and can be modified by you. It is how you will interact with opus. You can add or remove tool bars, customize the view and save in your user folder which is named with your Cornell user I.D.
  - Note that the status light in the bottom right hand corner of OPUS is green. If the light is red there is a problem with the microscope or spectrometer that needs to be corrected before taking measurements. Please contact the facility manager.
  - Opus has an excellent help menu. Almost every OPUS Dialogue contains a help button which leads you directly to the appropriate online description. This feature can be extremely helpful.

To set up for a measurement go to: Measurement > Advanced Measurement

- **Basic tab**
  - Be sure that you have entered in a sample description and sample form. They will be used to name your files when you save them. Files will be saved in C:\Documents and Settings\Administrator\Administrators documents\FTIR Users\"Cornell ID"
  - The file will be saved automatically with the following convention: Sample Description_Sample form_Date. Sample Description and Sample Form can be modified on the advanced tab in advanced measurement. For example a spectrum with a sample description of "polyethylene" and a sample form of "film" taken on 7-11-2011 will have the file name of polyethylene_film_7-11-2011.
  - If files use the same name they will be appended with .1 .2 .3… .x
  - File name and File path on advanced tab should not be changed as they will populate automatically using the guidelines stated above.
- **Advanced Tab**
  - Do not change the File name or path on the advanced tab!
  - Resolution: This should generally be set to 4 cm^{-1} If you have a need for greater resolution please ask me and we can go through it together.

- Fill in the Sample scan time, Background scan time. This can be entered as a time or as a specific number of scans. Sample and background scan time do not need to be the same. If you have a weak signal you can increase the scan time which will allow for greater exposure to the detector. Also increasing the scan time will increase your signal to noise ratio.
- Enter spectral range for saved data and the results spectrum type that you would like to view your data in. Also select the data-blocks that you would like to save

- **Optic tab**
  - External Synchronization
    - This should be set to off
  - Source setting
    - This should automatically update when source is changed
  - Optical filter setting
    - Generally should be open
  - Aperture setting
    - Adjust this to increase or decrease the signal strength. Also preamp gain can be adjusted to raise or lower the signal strength.
  - Measurement Channel
    - Set to Right exit when using the microscope and sample compartment when using the spectrometer.
  - Background meas. Channel
    - This setting should be the same as the measurement channel
  - Detector setting
    - The MCT detector is located in the microscope; it can only be used in conjunction with the microscope. If using this detector measurement channel and background channel need to be set to right exit. This detector has a spectral range of 12,000 – 600 cm^{-1} and a sensitivity of $2x10^{10}cm\ hz^{½}\ W^{-1}$
    - The DTGS detector is located in the spectrometer; it can be used only with the spectrometer. If using this detector the measurement channel and background channel should be set to sample compartment. This detector has a spectral range of 12,000 – 250 cm^{-1}; and a sensitivity of $4x10^{8}\ cm\ hz^{½}\ W^{-1}$.
  - Scanner velocity
    - This should be set to 20kHz for the MCT detector and ≤10kHz for the DTGS detector.
  - Sample signal gain
    - Leave at Automatic

  - Background signal gain
    - Leave at Automatic
  - Delay after device change
    - Change as needed
  - Delay before measurement
    - `Change as needed
  - Optical bench ready
    - Should be set to off
- **Check Signal tab**
  - The check signal tab allows the user to view the sample spectrum and the interferogram in real time. It is recommended that you check this tab prior to taking your spectrum. The show box located at the lower left of GUI allows you to toggle between the interferogram, spectrum, and the ADC count. The ADC count allows the user to monitor the Amplitude and zero-crossing position from a distance. The user also has the ability to scale the display.
- **Saving .xpm file**
  - Once the measurement is set up you can return to the advanced tab and save the measurement. The measurement can be saved in your user folder; it will be saved with the file extension .xpn. An .xpn file can be created for different types of measurements. To save go to the advanced tab and click the save button. Enter the name for the file and ensure that the file path points to the proper user folder and save.

Note: The following tabs are for advanced measurements contact the facilities manager if you would like help using these settings.

- **Acquisition tab**
  - **High and low frequency**
    - These limits can be adjusted to a lower bandwidth but the high pass and low pass filters must also be set to filter all wavelengths outside of the high and low limits.
  - Acquisition mode
    - This should be set to double sided forward-back
  - Correlation mode
    - Correlation can be used to perform a data integrity check
- **FT tab**
  - Phase resolution

        - This should be set to provide at least 250 phase interferogram points.
    - Phase correction
        - Should be set to Mertz
    - Apodization function
        - Should be set to Norton and Beer med for most applications. If you are trying to attain a very high resolution you may want to consider other functions.
    - Zero filling factor
        - This will smooth your spectrum. The higher the number the greater the smoothing
- **Display tab**
    - Use to scale the display
- **Background tab**
    - OPUS will use the most recent background spectrum taken when calculating your sample spectrum. If you would like to use a background spectrum that was taken previously you can load it on this tab and OPUS will use it.

- **Transmittance Measurement 15x**
    - **Microscope**

1. Activate viewing mode on the front of microscope.
2. Activate the transmittance mode.
3. Place the sample on the stage and position it in the optical path.
4. Swing the 15x objective into the optical path.
5. Select the light path for sample viewing (route light to the binocular).
6. Focus on the sample. Using the visible polarizers can help in defining regions of interest.
7. Adjust the image contrast by closing the Köhler aperture for the transmittance. The black plastic ring below the condenser.
8. Find the area of interest by moving the stage in the x-y plane.
9. Define the sample area by using the knife edge aperture.
10. Remove the sample from the optical path. Make sure that the detector doesn't oversaturate and maintain the knife edge aperture in its current position.
11. Swing the alignment aperture into the beam. Adjust the condenser in the x, y, and z directions until the image of the alignment aperture is in focus and in the center of the reticle.
12. Be sure that you have opened the Köhler aperture.
13. Start the reference measurement.

14. Once reference measurement is complete move the sample back into the optical path.
15. Readjust the condenser in the z direction see step 13.
16. Start the sample measurement.

- **Spectrometer:** Note the spectrometer can only take measurements in transmittance mode using the DTGS detector. Specimen must be sufficiently thin to allow transmission of IR light (<50µm).

1. Set up .xpn file in OPUS
2. Acquire background spectrum
3. Place specimen in sample compartment
4. Acquire spectrum

- **Reflectance Measurement 15x**

1. Activate viewing mode.
2. Activate reflectance mode.
3. Place the sample on the stage and position it in the optical path.
4. Swing the 15x Objective into the optical path.
5. Select the light path for sample viewing.
6. Adjust the light intensity starting from a low intensity level.
17. Focus on the sample. Using the visible polarizers can help in defining regions of interest.
7. Adjust the image contrast.
8. Find the sample area of interest by moving the stage in the x-y plane.
9. Define the sample area using the knife edge aperture.
10. Remove the gold mirror from its case and exchange the sample for the supplied gold mirror.
11. Focus on the mirror edge or an existing scratch on the mirror.
12. Activate the IR mode.
13. Start the reference measurement.
14. Once reference measurement is complete, exchange the mirror for the sample.
15. Activate the viewing mode.
16. Focus on the sample.
17. Find the sample area of interest by moving the stage in the x-y plane.
18. Activate IR mode.
19. Start sample measurement.

- **Capabilities and notes**
  - Measurements in transmittance mode are suitable for very thin specimens (<50µm)
  - Diameter of ATR Crystal tip ~100µm.
  - Refractive index of ATR Crystal = 4. Sample must have a refractive index that is < ATR crystal.
  - Microscope measured area: Optimized for diameter of 250µm; minimum diameter of 20µm with standard objective
  - Spectral range:
    - Beamsplitter: 10,000 – 400 cm^{-1}
      - Note: it may be possible to attain measurements in the 11,000cm^{-1} range. Contact facility manager if you are interested in measurements in this range.
    - Mid band MCT detector: 12,000-600cm^{-1}
    - DTGS detector: 12,000 – 250cm^{-1}
  - Resolution: Better than 0.9cm^{-1}.
  - Wave number accuracy: better than 0.019cm^{-1} at 2,000 cm^{-1}
  - Note: if the x-y stage joystick fails to move the stage go to the measure menu and select motorized stage control. Toggle the joystick control check box. The mechanized stage should now be active.