1. General Procedures at U2B Beamline

1.1. Set up your experiment

Infrared U2B beamline is equipped with three sample compartments, several windows, detectors and beamsplitters. Depending on the frequency range you need, choose appropriate beamsplitter, detector and window at the end of the synchrotron tube. See the following chart:

Detector	Freq.range (cm^{-1})	Beamsplitter	Window
MCT/A	4000-750	KBr	KBr
MCT/B	4000-450	KBr	KBr
DTGS/PE	700-10	Solid substrate	PE
Si:B	3000-100	KBr, solid substrate	KBr, PE
Bolometer	700-10	Solid substrate	PE

If you need to change the window at the end of synchrotron tube, contact PRT staff.

A: Beamsplitter change

- Turn spectrometer off; Open small door at the back-right corner of the spectrometer; Locate beamsplitter in use, i.e. in the optical path; Turn the handle-lock 180 degrees counterclockwise to unlock it. Take the beamsplitter out grabbing it by the handle. **Never touch optical parts!**
- Place the beamsplitter into its holder at the far right of the spectrometer; take the one you need from the holder, and place it into the optical path. Turn the handle-lock 180 degrees clockwise to secure it. Turn spectrometer on.

B: External sample compartment / external detector setup

MCT/B detectors and Si:B Photodetector/Bolometer dual detector system need to be connected to the spectrometer through a special Detector Interface Box. The box also instructs the spectrometer where (i.e. behind what sample compartment) the detector is located.

- **Turn Spectrometer off.**

Warning: The spectrometer is very sensitive to connecting and disconnecting cables. If cables are connected while it is on, the spectrometer may experience complete internal failure, or its motherboard may die, which only a Nicolet technician can repair.

- Locate Detector Interface Box.
- If you need to change the sample compartment through the Detector Interface Box, remove the screws that secure the cover using a Phillips screwdriver. The screws are located on the bottom of the box. Turn the box right-side up and lift the cover off.
- Find the bank of switches labeled SW1 on the left-back corner of the box. Set the switches according to the position of your sample compartment containing the detector, using the following table:

Detector Location	*Address*	*SW1 Setting*			
		1	*2*	*3*	*4*
Right-side µscope	D5	Off	On	On	On

Left-side AEM	D6	On	Off	On	On

- Place the cover back on the interface box, secure it with the screws and connect it to the detector via a BNC cable.
- If you need to change the type of the detector, locate the jumper block at the back of the Detector Interface Box. Set the jumpers according to the type of the detector, using the following table:

Detector Type	*Install shorting blocks on JP4*
MCT/B	Pins 5-6 and 7-8
Bolometer	Pins 1-2 and 5-6
Photoacoustic (use for Si:B photodetector)	Pins 5-6

- **Turn spectrometer on**.

1.2. Cool the detector

1.2.1. Intenal DTGS detector with polyethylene window (in the main bench) needs no cooling.

1.2.2. Si:B /Bolometer dual detector works at liquid helium temperature. Find the separate instruction sheet on detector pumping, pre-cooling and filling with LHe.

1.2.3. Mercury Cadmium Telluride (MCT) detectors work at liquid nitrogen temperature. Four of them are in use at the beamline. Find the one you will use (narrow-range MCT/As are internal, located in the main bench and in the μscope; wide-range MCT/Bs are external detectors) and fill it with liquid N_2 (LN2) before use.

- The 5-liter dewar flask can be found under the spectrometer's bench. If empty, fill it from the LN2 cylinder located under the stairs to U4IR, or contact staff for help.
- Find the plastic funnel with aluminum tubing, located in the black cabinet next to the spectrometer. Lift the small plastic cover covering the internal detector (at the left front end of the spectrometer or at the top of μscope). Put the funnel into the hole of the MCT detector (internal MCT/A detector in the spectrometer and MCT/B detectors) or directly into the opening (internal μscope MCT/A). Slowly pour LN2 until some of the liquid starts to spill around the detector.
- Allow the detector to settle down for at least 10 min before collecting spectra.

Note: Bench Status in OMNIC program indicates the status of the detector. Green check mark indicates that the detector cold, while yellow circle indicate that the indicator is still warm. If you see a red cross, there is a problem in bench/computer communication - contact U2B personal for assistance.

1.3. Create folder for your spectra

- Turn on the monitor. (The computer should be on at all times.)
- Create a folder for your data: from the desktop, double-click on My Computer - (C:) – Users. If there is no folder named after you or your principle investigator, create one by clicking on File – New – Folder. Name your folder. Then, double click on it, and create a subfolder for this run by clicking on File – New – Folder. Name the folder after current month and year (for instance, May01).

1.4. Set your Parameters

- From desktop, double-click on OMNIC icon on the computer.
- Click on Edit – Options.
  Click on File tab. Click on Initial Spectra, Select Path, (C:) – Users – Your_Folder – Your_Subfolder.
  Check Warn if aperture too large to attain resolution push-box.
  Click on Save and Exit.
- Click on Collect – Experiment Setup.
- Choose Collect tab.
  Number of Scans: usually in power of two: 32, 64, 128, 256 etc.
  Tip: Signal-to-noise ratio depends on square root of number of scans. Try 64 scans first, increase if necessary.
  Resolution: choose from the pull-down menu (ranges from 0.125 to 32 cm^{-1}).
  Tip: A reasonable resolution for liquid and most solid samples is 2 or 4 cm^{-1}. Higher resolution is impractical as it results in longer times to collect data with the same number of scans.
  Final Format: depends on the experiment. For transmission mode, choose interferogram, single beam, % Transmittance or Absorbance. For reflection mode with microscope, only % Reflectance and log (1/R) are allowed.
  Correction: unnecessary for initial spectra.
  File handling: click on Save Automatically or Save Interferograms, and type a four-character-long base-file-name.
  Background handling: click on Collect Background after ……. Minutes.
  Tip: Type in a high number of minutes if you do not want to be prompted each time for the background scan.
- Choose Bench tab.
  Gain: set to Autogain.
  Velocity: typically choose 3.1647 for MCT detectors, 1.89 or slower for Solid Substrate DTGS (Far IR) and Si:B detector/Bolometer.
  Tip: Higher velocities of the scanning mirror may reduce the throughput.
  Aperture: affects only the IR beam from the internal globar source.
  Tip: use aperture 32 and screen B for main chamber with air in the optical path.
  Click on *Peak to Peak*.
  Sample compartment: Click on Main for internal spectrometer compartment, Right μSope, %T for microscope in transmission mode, Right μSope, %R for microscope in reflection mode, or Left AEM for Far-IR external vacuum chamber.
  Detector: If you are using an extenal detector through Nicolet Detector Interface Box, detector will be set automatically. Otherwise, choose MCT/A for mid-IR MCT, DTGS polyethylene for Far-IR DTGS detector.
  Note: Bolometer detector will be shown as Ge.
  Beamspliter: Click on KBr for Mid-IR, Solid Substrate for Far-IR.
  Source: Click on IR for internal global source, External for synchrotron light.
  Accessory: Valid only for the main chamber; choose one from the pull-down menu, if it is used. Otherwise, leave None.
  Window material: Choose from the pull-down menu, if it is used. Otherwise, leave None.
- Click on Quality tab.

Unmark Use spectral quality checks box.

- Click on Advanced tab.
  Zero filling: If not an advanced user, leave none.
  Apodization: Choose from the pull-down menu.
  Tip: Choose Happ-Genzel or Triangular. Do not use Boxcar.
  Phase Correction: usually Mertz.
  Set: Sample spacing based on spectral range
  Set: Set filter based on velocity
- If you see the peak (centerburst) of the interferrogram, click on OK. You do not need to enter in Diagnostic tab menu.
  If no peak is visible, try resetting the bench from Diagnostic tab menu; see Troubleshooting section.

Note: Scan LED diode on the spectrometer indicates whether the spectrometer is scanning or not. Observe it after you click on Rest Bench button.

1.5. Save Parameters and Record throughput

- Click on Save As.. push-button, type in a name of the parameter file and press Enter, then click on OK push-button to exit from the Parameter menu.
- Open U2B Logbook and fill the following information in the table provided:
  Date, Sample compartment (main / external / microscope), Attenuating factor (screen B for main compartment / lens objector for microscope), Substrate (air / gold / reflective coating), Aperture (if using internal source), Final Format (%T / %R), Current (for synchrotron source), Peak to peak signal voltage and User name(s).

1.6. Collecting data

- Place the accessory filled with the material you want for the background in the optical path (liquid cell filled with supporting solution, KBr pellet, bare window or reflective slide, etc.). Depending on the Final Format chosen, click on Collect – Background or Collect – Sample for Absorbance or Single Beam Spectra, respectively.
- When the background scan is done, place the sample in the optical path. Click on Collect – Sample.
  Tip: To break scan, click on Stop button, then close the window by a click on X in top right corner.

1.6. Troubleshooting

- The connection between the spectrometer and the computer is established after the spectrometer is turned on and starts scanning (usually within one minute). Click on Collect – Experiment Setup – Bench, and verify that the correct detector/detector location/ beamsplitter/source are chosen for your experiment. If you do not see any signal (i.e. centerburst), make sure there is nothing in the optical path towards the detector, and the detector is on (if external) and properly cooled (if using LHe or LN2 cooled detector). Then try to Reset bench by clicking on Collect – Experiment Setup – Diagnostic – Reset bench.
- If using microscope, try focusing again. Make sure you are focused through 32x IR condenser.
- If the above procedure fails, try it once again. If you cannot get any signal after the second try, **call U2B staff**.

2. Working with Internal Sample Compartment

Only transmission spectra can be recorded in the internal sample compartment at this time. A simple procedure is described below:

2.1. Assemble liquid transmission cell

Spectra-Tech or International Crystal Labs Demountable Cell Kit need to be assembled according to the instructions booklet. General instructions follow:

- Put a Teflon Gasket into Back Plate, then place an undrilled Window. Three sets of window materials are provided: CaF_2, BaF_2 and Quartz windows. Place a spacer on top. Carefully align the drilled window, Teflon gasket and Needle Plate. Place the Teflon O-ring on top and carefully screw in the Knurled End Cap.
- Using two syringes, fill the cell with the supporting solution.

2.2. Take the Spectrum

- Collect data as described in section 1.6.

3. Working with Microscope

3.1. General

- Verify that beige plastic tubes are placed between the spectrometer bench and the microscope. They are needed for purging.
- Remove the protective glass slide from the sample table.
- Select *Transmission (%T)* or *Reflection (%R)* mode
- If you need to use Si:B photodetector/bolometer dual detector system, review the guide for filling and work with the detector.
- Call PRT staff for the training on the detector filling.

3.2. Focussing

3.2.1. Stage controller

- Turn on the mapping stage controller, located on the right side of the microscope. The power switch is on the rear right corner of the unit. Lift the view-screen up. The sample table starts moving.
- After the initialization of the mapping stage controller is complete and the table stops moving, press 1. Then move the sample table until the region of interest appears in the viewer by moving the handle of the stage controller. You can move it left, right, up or down.

3.2.2. Pre-focussing

Pre-focusing is best done with no aperture set in the optical path, and using the 10x optical lenses:

- Take both apertures away from the μscope.
- Rotate the lenses until the 10x optical lens snaps into place.
- Push *View* button (located on the bottom of the microscope) on. Green LED indicator will lit.

Tip: View button will not lit if you are in Experiment Setup screen. Close Experiment Setup.

- Turn either bottom or top light slide control knobs (located on both sides of the *View* button) about half way.

Tip: In transmission mode, use bottom (left) light control knob; for reflection, choose top (right) light control knob.

- By pulling out the slide knob on the right topside of the microscope, choose where you want to see the image: through the eyepiece (Position A), camera (C), or both (B).

Tip: Image may appear clearer through the eyepiece.

- If you choose to look at the image through the camera, turn on the Imaging program on the computer. From Desktop, click on Camera icon. Turn on the camera by pushing the red button on the camera power supply unit.
- Turn the coarse height-adjustment knobs, located on both sides of the microscope until the image appears clear in the viewer. Focus using the fine adjustment knobs.

Note: The fine height knob on the left side is calibrated so that each division corresponds to one micron in height when using 32x lens condenser.

- Once you found the spot you want, lower the sample stage all the way down using coarse adjustment knobs and rotate the lenses until the 32x IR lens snaps into place. Then focus on the sample again, as explained below.

3.2.3. % T mode:

- Place LOWER (1.0 mm) aperture in the gap just under the bottom lens. Place 32×UPPER (3.2 mm) aperture at the gap just above the upper lens.
- Insert a colored glass into the gap at the top of the microscope.
- Turn on the top light control knob. A *colored* circle appears. Adjust the sample plate height so that the colored circle appears clearly. Then turn the top light off.
- By rotating metallic knob on the right side of the microscope, bring the bottom lens to the sample table as close as possible.
- Verify that the bottom light is on. Move the lower lens down by rotating the metallic knob until a clear *white* circle appears clear in the viewer.
- Turn on the light of upper lens; the *colored* circle appears again.
- Adjust two screws at the two rear edges of the sample table to overlap the two circles.

Note: If the light passes through a window (or windows) in the optical path, you need to compensate for the thickness of the window(s). The compensation knobs are located on each objective lens and have markings 1, 2, 3, … The numbers represent the thickness of your window in mm. Set them accordingly.

3.2.4. % R mode:

- Put the gold sample plate or a reflective slide, onto the stage.
- Remove the 32×UPPER, or verify that it is removed.
- Find a bright spot on the gold/reflective slide surface by moving the handle at the stage controller.
- Put the 32×UPPER back into its place.
- Move the stage plate as close to the upper lens as possible.
- Focus by moving the sample table down until the light circle is clear.

3.3. Infrared Mapping

3.3.1. Calibration

Note: Calibration of the Video camera is very sensitive operation. **Do not** attempt to do it without staff supervision.

- Turn off camera program, or verify that it is off.

Note: Atlμs and the Camera demo program may interfere with each other because both use the same video camera.

- From OMNIC start page click on Atlμs. Atlμs window opens.
- Click on Image, then Create Calibration. With 32x IR lens, look through the eyepiece at the image you want to map. Move the image until it is centered in the eyepiece. Save the image by clicking on Save As…, and give the name of the file.
- Click on Calibrate Video.
- Two persons are needed for this operation. Look at the image at the monitor screen. If it is off-centered, unscrew the set screw on the camera and lift the camera to expose three screws that hold the camera adjustable plate. Loose the screws.

- Put the camera back into its place. Grasp the camera and the adjustable plate and move it so that the image appears centered at the screen. Hold the camera still while the co-worker fastens the camera adjustable plate screws. Fasten the set screw at the camera.
- If the image at the monitor is still off-centered, you need to reset it again, starting from the point No. 4.

3.3.2. Mapping parameters

- From OMNIC start page click on Atlμs. Atlμs window opens.
- Click on Area Map Tool button (square) and drag a box around the image you want to map. If you want a line map, click on Line Tool button (diagonal line) and draw a line across the image.
- Click on the Background Point Tool button (cross with a B). Point to an area where you want the background taken.
- Click on Collect – Map Setup. The start and end position of the map, as well as the background coordinates should be already set. Check Step size and Number of Steps.
- Click on Aperture tab. Choose Rectangle with 10 x 10 μm. Click on Apply. A red square should appear in the viewable window. Place the adjustable aperture at the top slot. Move the aperture screws to set a square of 10 x 10 μm that overlaps with the red square.

Note: You can also set it through the eyepiece; remember that each division in the eyepiece ruler is 2.7 mm long.

- In the Collect tab, click on Map Title, and give the title to the file. Set Number of scans, Resolution, Apodization and Final Format according to section 1.4.
- Click on Options tab. Check the boxes next to Save Video Frames in the Map file and Store Map with relative coordinates. Check background options.

3.3.3. Mapping Measurement

- In the OMNIC window, click on Collect, and set the parameters (see section 1.4). Be sure to set the Source as *External* for the synchrotron beam.
- Focus the microscope for measurements in transmission or reflection mode (see section 3.2).

Note: Rotating the fine height-adjustment knob may enhance the throughput.

- In Atlμs window, click on Collect - Collect Map. The measurement begins with collecting the background scan first.

Note: Depending on the number of scans, resolution and step size, it may take from several minutes to several hours to complete the measurement. If possible, start the experiment just after the beam is refilled to avoid lost points during the injection.

4. Working with External cryostat box

The procedure for the external box is experiment-specific. Only general information can be summarized here.

Note: The spectrometer needs to be purged extensively if any part of the purge in the beam-path is broken. This is crucial for far-IR experiments. Therefore, a PRT member should perform the change of the window at the end of the synchrotron tube and the alignment of the beam into the External Box and prior to your arrival. Do not attempt to open spectrometer or any part of the beam path without PRT supervision.

- Visually inspect the External box and notify the PRT staff immediately if you notice anything that may interfere with your experiment.
- If you need to use Si:B photodetector/bolometer dual detector system, review the guide for filling and work with the detector.
- Call PRT staff for the training on the detector filling.
- Verify that the appropriate detector is placed at the end of the External box and connected through the Nicolet Detector Conversion Box (see procedure described in section 1.1).
- Turn the detector on.
  - The Si:B photodetector is powered on by plugging it into the wall outlet through a transformer only.
  - The bolometer is either powered by the same transformer, or through batteries. Check with PRT staff what you will be using. If powered by batteries, you will be responsible for changing batteries every 48 hours.
- Turn on all preamplifier switches at the side of the preamplifier box, labeled Si:B or bolometer, depending on which detector you are using. Set the gain on the detector on setting No. 3 for Si:B, or 1000 for bolometer.
- Perform the procedure described in Section 1-3 to 1-5. Make sure that Collect – Experiment Setup – Bench shows correct compartment (Left AEM), source (external) and the correct beamplitter.
- Observe the interferrogram centerburst. If none is detected, refer to the Troubleshooting section (1.6).