Figure 2.1: Photograph of the 1.4.3 FTIR spectromicroscopy endstation.

2. Beamline 1.4.3

Using the IR Microscope

The FTIR instrumentation for beamline 1.4.3 is shown in Figure 2.1 and consists of a Nicolet 760 FTIR bench (to the left), a Spectra-Tech Nic-Plan IR microscope (in the center), and video display and stage controller for the microscope (on the right). One of the primary reasons for doing infrared spectroscopy at a synchrotron light source is the large enhancement in brightness (flux per unit area). This brightness advantage manifests itself most beneficially when focussing the light to a very small spot size. We have been able to achieve essentially diffraction-limited spot sizes in the mid-IR using the Nic-Plan microscope with the ALS source. Figure 2.2 demonstrates the tight focus achieved using the synchrotron source, the 32x objective, and measuring the transmission through a 5-micron pinhole on the sample stage. No other apertures were used. The pinhole was moved via the computer controlled x-y stage to map out the extent of the beam profile. The cross sectional Gaussian widths of the spot were 6.2

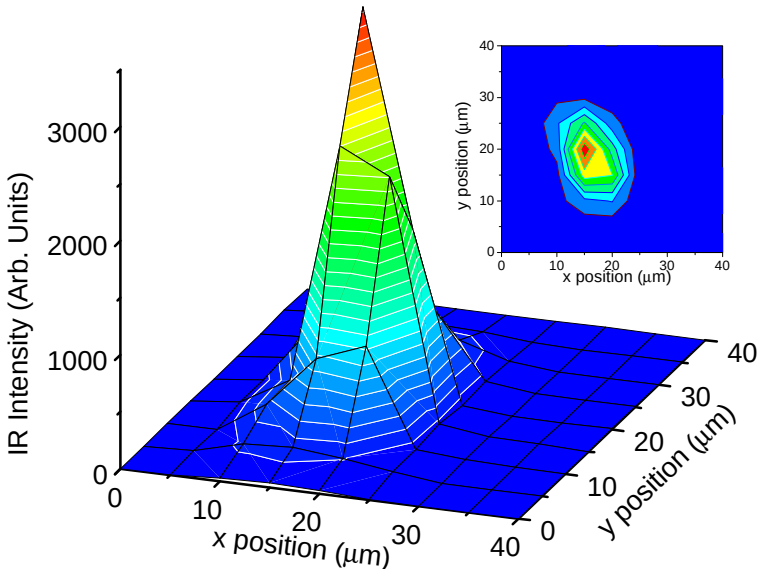


Figure 2.2. Measurement of the IR spot size with the synchrotron as the light source.

by 10.1 microns. Since the mid-IR wavelengths used are centered in the 3 to 10 micron range, and the objective lenses have a numerical aperture of approximately 1, we would expect the diffraction limited spot size to be approximately the wavelength of light, i.e. around 10 microns or better. This has been achieved on BL1.4.3. When the same measurement is made using the internal Globar™ IR source, the spot widths are 80 by 100 microns! Thus while the synchrotron source does not provide a huge gain in flux compared to the Globar™, we are able to get ALL of the light from the synchrotron through a 10-micron aperture. We routinely measure a signal enhancement with a 10-micron sample of a factor of 200.

The collimated synchrotron light beam exits the switchyard at its lower left (see Figure 1.8) and then enters the beamline 1.4.3 hutch through a hole in the wall as pictured in Figure 2.3 below. The light is reflected behind the Nicolet 760 bench all in rough vacuum. The beam exits the vacuum via a KBr crystal window and then enters into the side of the Nicolet bench. Once inside the bench, the light goes through the FTIR Michelson interferometer, then is sent on to the sample which can be either in the main bench's sample compartment or in the Nic-Plan IR microscope, and finally to an IR detector. As the microscope is where large advantages are gained from coming to a synchrotron IR beamline, this manual will concentrate on describing the operations of the IR microscope.

When you first arrive at the beamline there are several things to do. The main FTIR bench and the IR microscope are always kept powered on. But the video camera, monitor, and microscope x-y stage controller will usually need to be turned on. The video camera is powered on by pressing the red button on its power supply as pictured in Figure 2.4. The video monitor's power switch is located below and to the right of the screen. And the x-y stage controller's power switch is on its backside, near the input of the power cable.

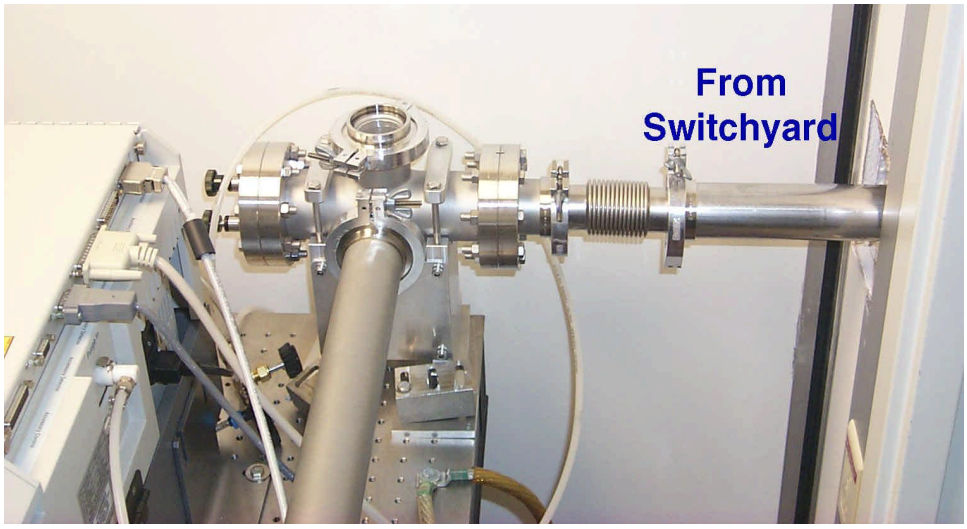


Figure 2.3. Photo showing the vacuum pipes transporting the IR beam from the switchyard to the Nicolet 760 for BL1.4.3.

Figure 2.4. Photograph of the microscope video camera power supply. To turn it on, press the red button on its front left and the red indicator light will illuminate to tell you that the power is on.

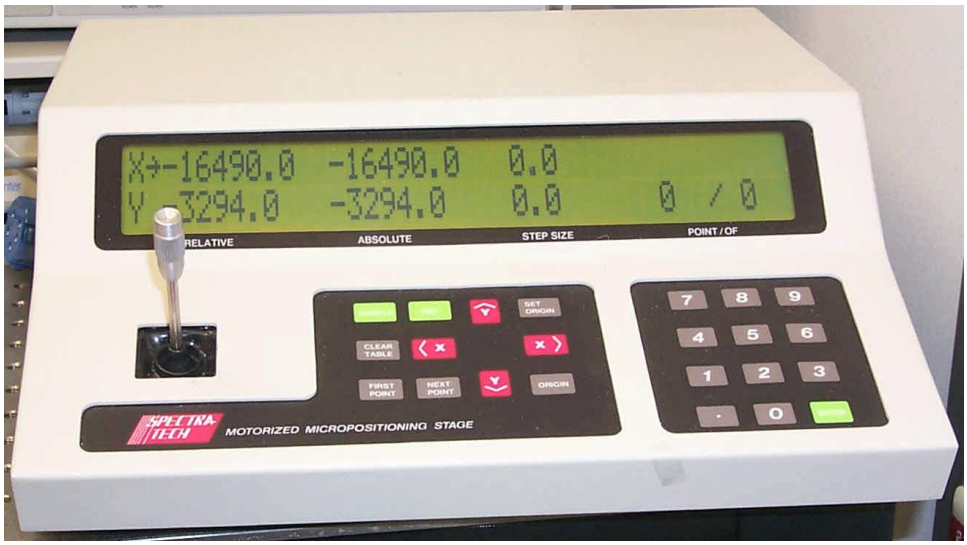


Figure 2.5. Photo of the microscope x-y stage controller. The power switch is on its back located near the power cord. When switched on you will be asked to make sure the stage is clear and press any key. Look to make sure the stage can have its full range of motion (there is nothing in the way of it moving both in x and in y), then press any key. The stage will now move to find its zero positions. The photo shows the controller in a typical operating mode. You can move the stage by using the joystick on the left. One x-y position can easily be memorized by pressing the SET ORIGIN button twice. Once this is done, pressing the ORIGIN button will take you to the memorized location. The numbers on the front display are in microns with relative being the displacement relative to the ORIGIN, and absolute being referenced to the center of the stage's motion.

The next task is to cool down the Mercury-Cadmium-Telluride (MCT) IR detector inside the microscope by filling its dewar with liquid nitrogen. A funnel is built in to the top right of the microscope (gray flip top on a blue plastic mount) and is connected to the MCT's vacuum-insulated dewar. Open the flip top, and *slowly* pour in liquid nitrogen. A green thermos labeled 'Nicolet' holds approximately the correct amount of liquid nitrogen to fill the MCT detector's dewar. When first beginning to fill, pour in only enough liquid nitrogen to fill the funnel (you will see it start to bubble against the plastic screen), and then stop pouring for at least 60 seconds and let that amount drain down the funnel. Repeat this two or three more times to fully cool down the funnel and tubing system. Then *slowly* pour the rest of the liquid nitrogen into the funnel making sure to never overflow the funnel. Store the empty green thermos face down on the ground.

Setup for Reflection Measurements

Place a reference sample on the microscope stage, for example a gold mirror, and move the x-y stage using the joystick on its controller to place this sample directly below one of the IR microscope objectives. Figure 2.6 shows a close-up of the sample stage area of the IR microscope. The microscope has two modes of operation. It can either be in IR mode or View mode, but not both. This ensures that there is never a chance for the alignment laser to be in the microscope when you are looking at a sample with your eyes. Press the 'view' button on the front of the microscope to allow visual inspection of the sample, and rotate the upper illumination adjuster to turn on the upper light source. Now look either into the eyepieces or at the TV monitor and adjust the focus (using the coarse and then fine focus knobs on the left of the microscope) looking for the brightest

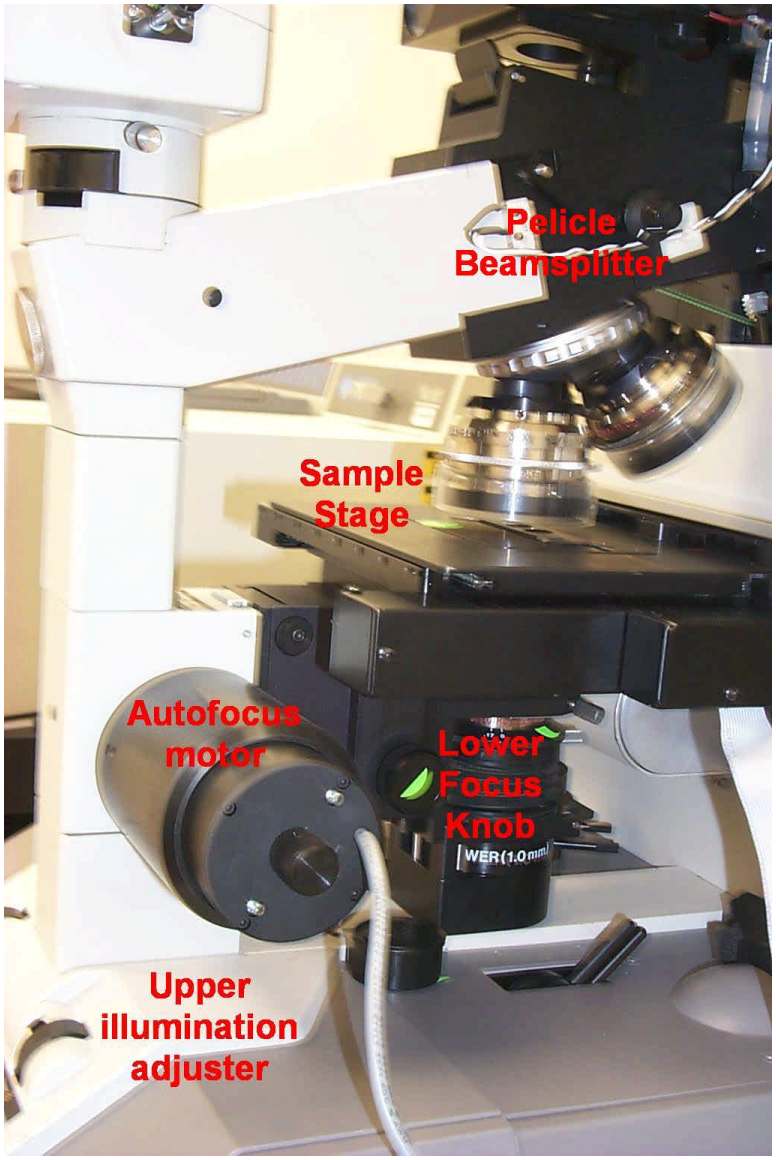


Figure 2.6. Photo detailing the sample stage area of the IR microscope with specific parts mentioned in the text labeled.

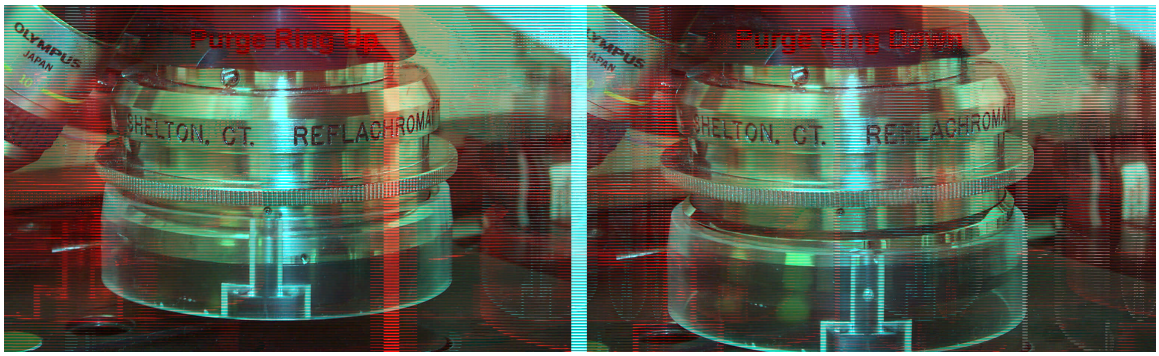


Figure 2.7. Detail showing how the purge ring is released and lowered around a sample. This will help to maintain a better-purged environment around your sample.

image. Using gold or another very good reflector will often require you to lower the upper light intensity when you are getting close to the correct focus before you can see the image as it will be too bright with the light at its maximum level. You can verify that you are focused on your sample by slightly moving the sample using the joystick on the x-y stage controller and you will see the image move. If it does not move, you have not yet found the sample focus.

Once the sample is in focus, you may want to lower the purge ring to help maintain the purge around your sample space, as demonstrated in Figure 2.7. The clear plastic ring is released by lifting slightly, rotating to place the pin in the middle slot and then dropping it down around your sample. While this is not perfect and will not work for all sample shapes and sizes, it will help to keep the purge more constant during your IR measurements therefore ensuring that extraneous water vapor and carbon dioxide absorptions in your measurements will be minimized. Make sure that the purge ring is placed back in its up position before you move your sample by any significant amount as the ring can get caught on parts of your sample or the stage when its moved by larger distances.

Now that the reference sample is in focus and the MCT detector is cooled, we can turn to the computer software running the spectrometer and making sure everything is working correctly. Skip to the OMNIC software section to continue.

Setup for Transmission Measurements

Place your sample on the microscope sample stage over a hole (the black three-holed microscope slides will work for this purpose, or any other holder you have that allows light to pass through the sample). If the sample is transparent to the eye then you can use it to align for transmission mode. If not, place your sample over one hole, and make sure there is another hole available on the sample stage for aligning. Adjust the stage such that the sample is directly below one of the upper microscope objectives and focus on the top surface of your sample, as described in the Reflection measurement setup above.

Once the sample is in focus, we now need to adjust the lower objective's focus to collect the transmitted light from the same location where the upper objective is focussing the incoming light. Insert the circular aperture with a label matching the objective you are

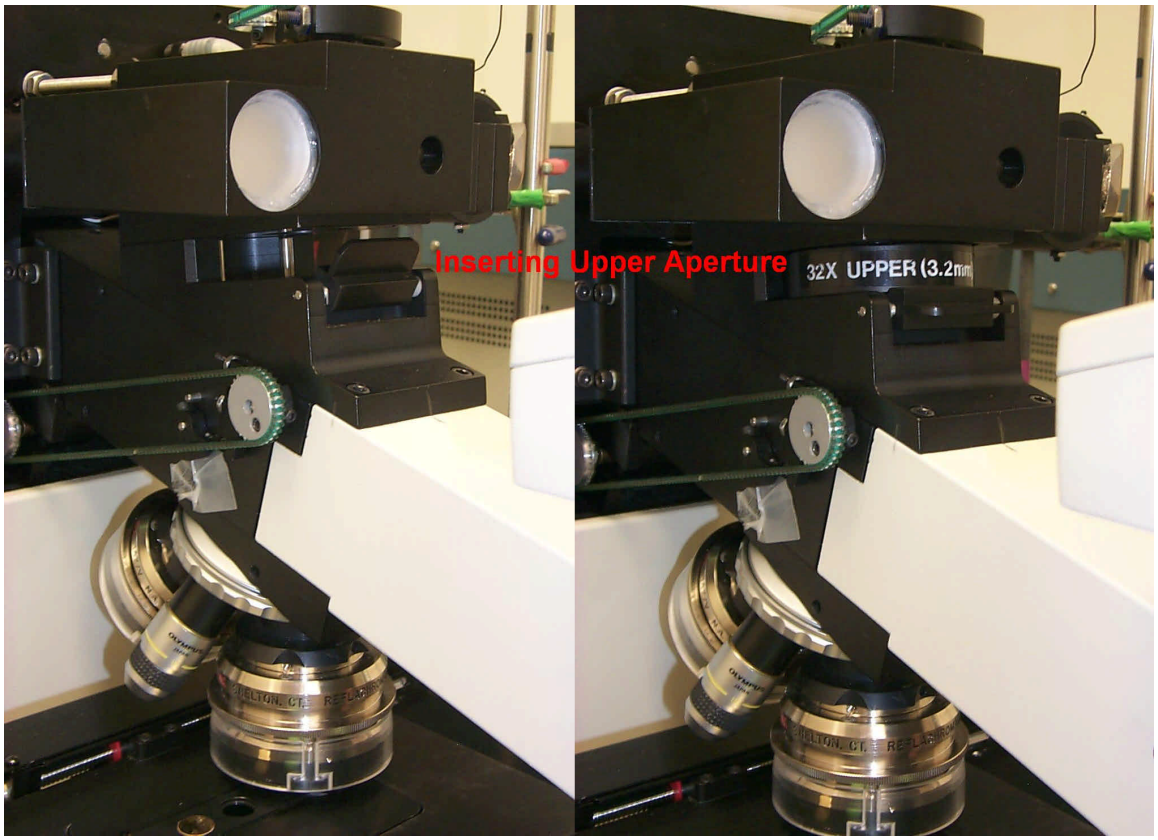


Figure 2.8. Photos showing where the upper aperture goes, and on the right with the aperture inserted. Make sure the label tape is facing out as the thickness of the tape would misalign the aperture if it is facing the alignment bars on the inside.

using (15x or 32x) into the upper location on the microscope as demonstrated in Figure 2.8. Lower the black plastic flap in front of the aperture location and slide the appropriate aperture into place taking care to have the tape label on the aperture facing out. This will define a 100-micron area on your sample that we will use to align the lower objective. You should now see the 100-micron area with the rest of your sample masked away on the television monitor or in the microscope eyepieces. *DO NOT ADJUST THE MAIN SAMPLE STAGE FOCUS KNOBS ANY FURTHER.* If your sample is not visibly transparent, press the SET ORIGIN button twice to memorize the sample position, then move the x-y stage to the open hole using the joystick on the sample stage controller, otherwise just leave the sample where it is and turn down the upper light source intensity.

Now insert the lower (1.0 mm) aperture into its position as shown in Figure 2.9. This time make sure the tape label is facing the rear of the microscope. This will again make a 100-micron area illuminated at the sample stage. Now increase the lower light source intensity, and focus the lower objective ONLY by using the lower focus knob as pictured in both Figures 2.6 and 2.9. Adjust this knob until you see on the television monitor or in the eyepieces as clear a circle of light as possible. Now turn up the intensity of the upper light source until you can just see the light reflecting off the top of your sample. If your sample was opaque and you are now over an open hole, rotate the pellicle beamsplitter into place using the knob pictured in Figure 2.6. This will allow you to see the light that

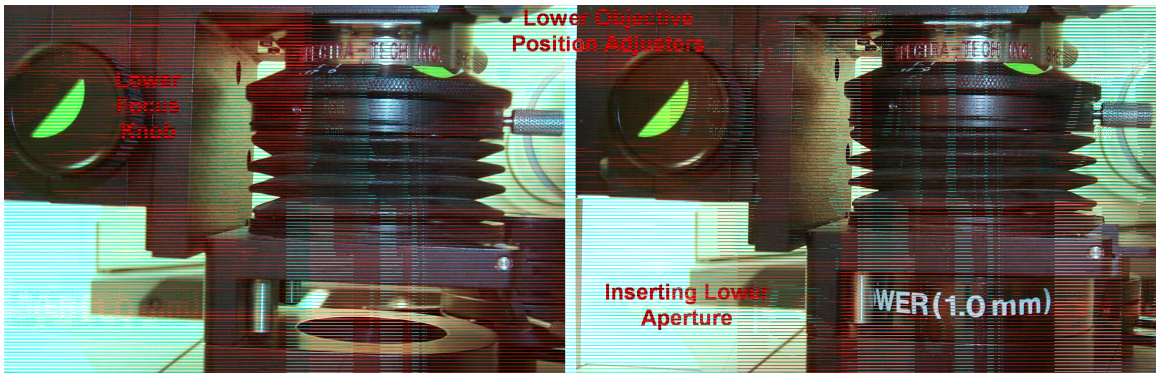


Figure 2.9. Close-up photograph of the lower objective showing where the lower aperture is inserted. The lower focus know is also seen and labeled (it has a piece of light green tape on it), and the lower objective position adjusters are barely visible at the top of the images.

is incident on the sample from the upper light source. Turn up the upper light source until you just see the image of the upper aperture.

The next task is to align the lower circle of light such that it is exactly at the same location as the upper circle of light. To help keep the two light sources straight, there are green and blue filters you can place in the respective light paths to color them differently, if you wish. The position of the lower circle of light can be moved using two ridged adjusters that are at 90-degrees angles from one another. One is barely visible and is labeled Lower Objective Position Adjuster at the top of Figure 2.9. These adjusters are not very linear and it takes some playing to get used to the way they move the lower aperture, but with a little playing you should be able to place the lower circle of light directly on top of the upper circle of light.

Now the upper and lower objectives are focussing on exactly the same part of your sample. If you had moved the stage to an open hole for the previous alignment procedure, you can now move the stage back to the location on your sample that you initially focussed on by pressing the ORIGIN button on the x-y stage controller. You can now move on to the OMNIC software section where we will check to make sure a good IR signal is being measured.

Nicolet's OMNIC Software

The software the runs the Nicolet spectrometer and the Nic-Plan IR microscope is called OMNIC. There are detailed manuals available for this software inside the beamline hutch as well as many online help topics and tutorials. Therefore this manual will simply give you an introduction to the basics of how to start up the software, setup your experiment, and acquire data. The software is capable of doing many types of data analysis and manipulation that will not be covered here, so the reader is referred to the OMNIC manuals and help menus for more information.

We will assume that you have a reference sample in the IR microscope, it is visually in focus, the MCT detector is cooled down, and you are ready to start a measurement. Start up the OMNIC ESP Software by double clicking on its icon from the desktop (shown at the right of this paragraph). You will be asked to input your user name. If you don't yet have a user name, please ask Mike to set you up with one. If you can't remember your user name, check the print out on the wall near the beamline computer.

Once you have entered a correct user name, OMNIC will start up and the main window will look similar to the one shown in Figure 2.10. To make sure the signal strength is good and to setup the spectrometer for collecting data, go to the Collect Menu and choose Experiment Setup. This will open a window like the one shown in Figure 2.11. The first two tabs, Collect and Bench, are the only ones you need to set up to collect data. The 'Collect' window is shown in Figure 2.11 and contains parameters for the data collection.

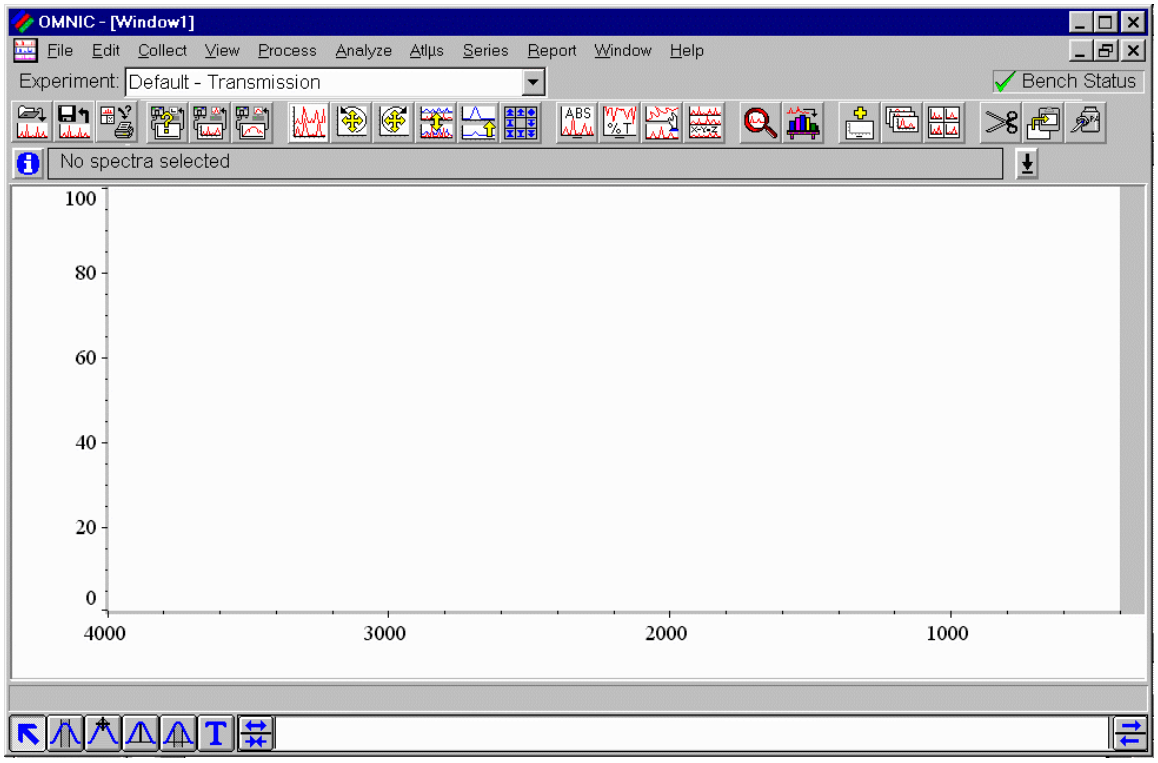


Figure 2.10. View of the main OMNIC software window.

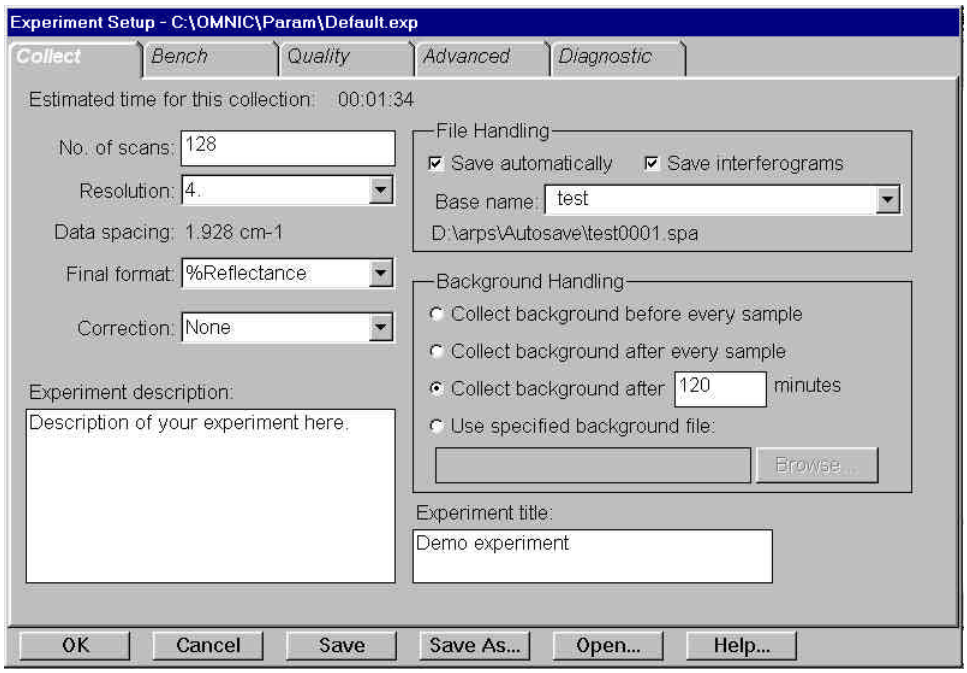


Figure 2.11. Experiment setup window with the 'Collect' tab selected. Typical values and selections are shown.

Set the number of scans to collect and co-add to obtain your spectrum (more scans will reduce any noise, but will add to the collection time). The resolution should be set appropriately for your sample. Most solid and liquid samples are fine with a resolution of $4cm^{-1}$, but check your data to see if any sharp peaks are limited by this resolution setting in which case you may want to increase the resolution to 2 or $1cm^{-1}$. The lower the resolution the faster the data collection time, so only use higher resolutions if you need to and are willing to spend more time collecting each scan. The data spacing value will be calculated automatically for you. The final format selected will be the way the data is processed and presented to you. For example, %Reflectance will automatically divide your sample spectra by the reference spectrum and present the results in percentage reflectivity. Other typical selections are Absorbance, %Transmission, and Single Beam (this last one does not divide by a reference spectrum). The correction selection can automatically correct for using an ATR (attenuated total reflectance) sampling technique and a few other corrections not typically used with the microscope. Usually leave this on None.

In File Handling, make sure to leave 'Save automatically' and 'Save interferograms' selected. This will automatically save each spectrum you acquire and by saving the interferogram you will have the ability to go back and reprocess your data using a different background or lower resolution at a later date. For Background Handling, most users have 'Collect background after 120 minutes' selected. This will remind you that after 120 minutes have passed that your background spectrum is getting old and you should really acquire a new one. Selecting either of the top two choices will force you to take a new background before or after *every* sample spectrum you take. This is usually impractical.

Finally, you can type in an experiment description and an experiment title that will be shown on the main OMNIC window. Once all the parameters are set (including the next

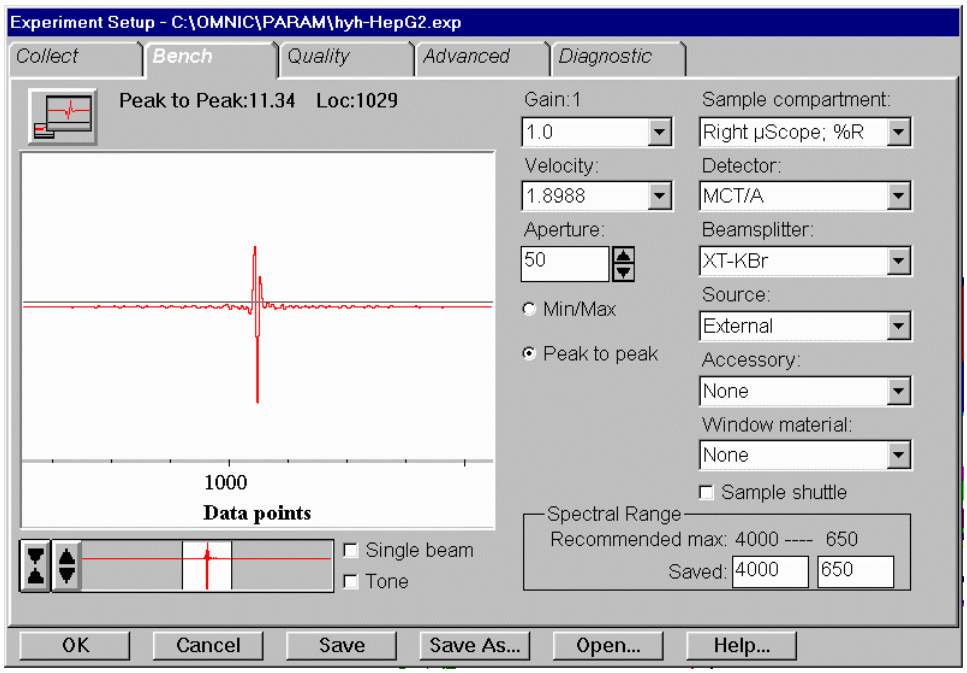


Figure 2.12. Experiment setup window with the 'Bench' tab selected. Typical values and selections are shown.

Bench tab), you can click on the 'Save As…' button at the bottom of the window and save the experiment file. Give the experiment file a name starting with your name or something you can easily remember so you can next time find the experiment file easily. Whenever you want to go back to the saved experiment setup, just open the Experiment Setup window, click on the 'Open…' button and select the experiment file you want to open.

The Experiment Setup window with the 'Bench' tab selected is shown in Figure 2.12. This screen shows the real-time interferogram signal as well as allows the user to do some spectrometer configuration setup. Assuming you have a reference sample in focus in the microscope and that the MCT detector is cooled with liquid nitrogen (from the previous sections), you should see a signal similar to the one shown in Figure 2.12. By adjusting the fine focus knob of the microscope, you should be able to maximize the signal intensity (the IR focus is slightly off from the visual focus). In reflectance mode with a gold slide as the reference sample, a peak-to-peak value between 8 and 12 on Gain 1 is expected if the system is lined up correctly. If you see a value much less than this, call Mike or Wayne to see if we can adjust the beamline alignment.

The parameters in this window are shown in Figure 2.12 with their typical settings. The Gain setting is a preamplifier gain that you can use to enhance small sample signals. The Velocity is the actual moving mirror speed in the interferometer as measured in cm/sec. If there is significant noise on the synchrotron beam, you may need to lower the speed to 0.9696 cm/sec, but usually a speed of 1.89 or 2.53 is fine. The Aperture is only used with the internal IR source, so you can ignore it. Down the right side selections, the Sample Compartment lets you select the microscope in either reflection (%R) or transmission (%T) mode, or the main 760 bench sample compartment. The microscope only has one detector, an MCT/A. The main bench is equipped with two detectors, a DTGS and an

MCT/B. The DTGS is a slow room temperature detector that is not fast enough to measure the synchrotron light pulses, but it can be used with the internal IR source. The MCT/B needs liquid nitrogen cooling, but it will work with the synchrotron source. There is only one beamsplitter available, a germanium coated KBr crystal. The Source selection lets you choose the synchrotron source (External), the internal IR source (IR), or the internal white light source (White light), which is used only for diagnostics. The last two selections, Accessory and Window Material are not used with our setups, so leave them set to None. Finally set the Spectral Range you want to save with your data. Using the synchrotron and the microscope, you can measure from 10,000 to about 550 cm^{-1}, but you can select a smaller range to match what you are looking for. A smaller range does not speed up the data acquisition, however, so it is suggested that you keep the range set to at least 4000 to 650 cm^{-1}, and larger is certainly fine too!

Once again, now that you have all these parameters set, you can click on Save As… to save this experiment file so you won't have to reselect everything next time!

The other three tabs in the Experimental Setup window are generally not used at our beamline. The Quality tab allows you to set up spectral checks during data collection; this is usually done only in industrial processes where the samples are very well known and you are looking for deviations from a standard. The Advanced tab allows you to set some of the Fourier transform parameters. The default setting will work for just about everyone, but Mike would be happy to tell you more about them if you feel you want to get to this level of understanding with FTIR. Finally, the Diagnostics tab window is shown in Figure 2.13. This is where to go when something is not working. The five blue icons along the top of the window indicate the status of the power supplies, alignment laser, internal sources, spectrometer electronics, and IR detectors, respectively. If there is a problem with any of these, a diagonal red slash will appear over the appropriate icon. If you see any of these indications, please call Mike or Wayne. If nothing appears wrong,

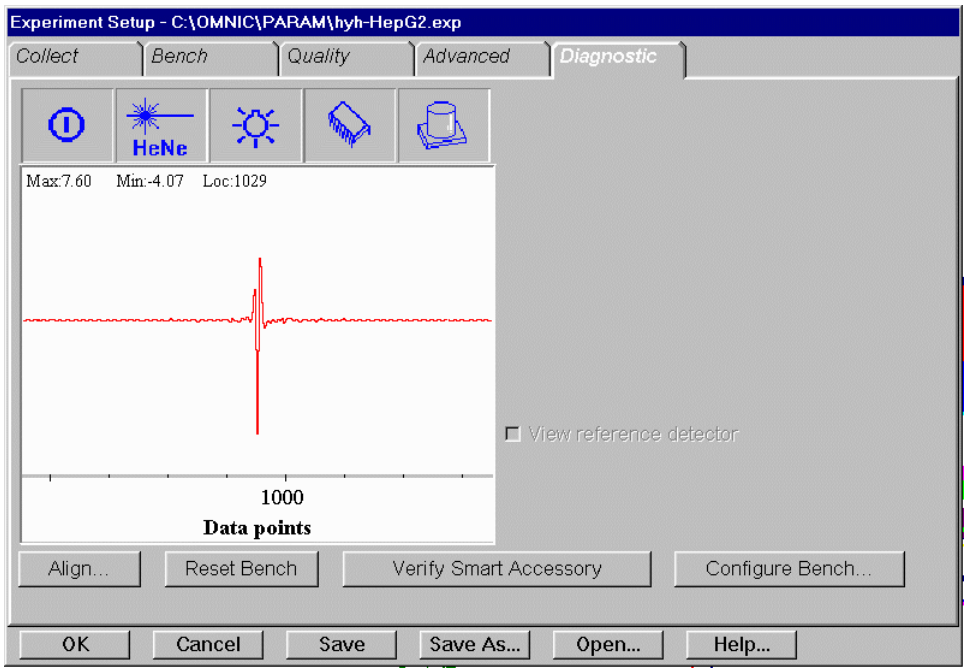


Figure 2.13. Experiment setup window with the 'Diagnostic' tab selected. The shown view is what you should see when everything is operating normally.

but you still do not see an interferogram signal, you may try pressing 'Reset Bench'. This will re-boot the bench electronics and will cause the spectrometer to re-initialize its zero positions. On occasion when the spectrometer has not had a signal in a many hours, this button will make the spectrometer find the zero peak mirror displacement again and start functioning like normal. If you continue to have problems getting a signal, please call Mike or Wayne.

Now that the Experiment Setup is complete, the data acquisition can begin! First you need to take a background spectrum. Once you are happy with the signal strength seen in the Bench tab of the Experimental Setup window, close that window by pressing 'OK'. Then in the main OMNIC window (Figure 2.10) select the Collect menu followed by Collect Background. The system may prompt you again to make sure you are ready, then it will acquire the number of scans you set in the Experiment Setup window. You can see the progress of the scans at the bottom left corner during data collection, and if you chose a resolution of 4cm-1 or poorer (larger numbers), the screen will show you an occasionally updated average of the data you are collecting.

Once the background is complete, you are ready to measure your sample. Put the sample in the microscope and focus on it visually (make sure you have pushed the View button on the front of the microscope). Then open the Experimental Setup window with the Bench tab selected and check the IR signal strength. Adjust the fine focus knob for maximum peak-to-peak signal, and if your signal is small you can increase the preamplifier gain value. When you are satisfied, close the Experimental Setup window by clicking 'OK'. Select the Collect menu followed by Collect Sample. If you are setup to automatically divide by the background spectrum (that is to say the final format is set to %Reflectance, Absorbance, %Transmittance, 1/R…), OMNIC will automatically present you with the final formatted resultant spectrum.

Now that you have an IR spectrum, you can do a lot of different types of data processing and visualization using the Process and Analyze menus. These functions will not be covered here, but can be found in the extensive on-line help files for OMNIC and the OMNIC manuals.

One note for when you are ready to print your data. Mike has made a new more comprehensive printout page for your use. The regular print function is still there, but it does not display a lot of useful information on the page, therefore I suggest using this new print button. There is a new print button on the toolbar that will open up this printout function, or just select Report/Preview-Print Report. You will then be asked to enter some optional text (anything you type here will be displayed at the bottom of the printout so you can use this for any notes you'd like to add to the printout. If you don't want to type anything, just leave it blank and hit OK.). The program will then show you a preview of what the printout will look like. If you are happy, select Print and it will be printed for you.

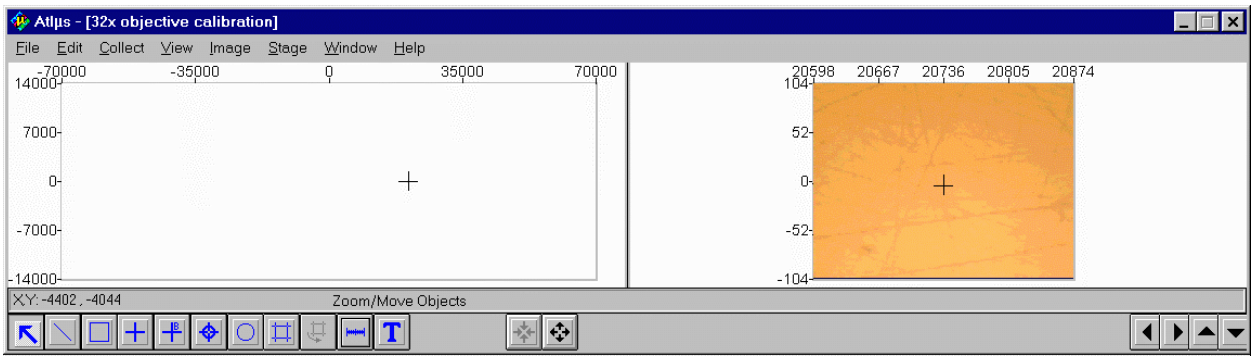


Figure 2.14. View of the main Atlμs window. The right side shows the live video image coming from the microscope. The numbers on the axes are the absolute position of the x-y stage in microns and they correspond to the numbers found on the front of the x-y stage controller (Figure 2.5). The left side shows the entire area the x-y stage is capable of going to, with a cross at the stage's present position.

Atlμs Mapping Software

The software that automates spectral mapping of samples in the IR microscope is called Atlμs and is written by SpectraTech. To launch Atlμs from the main OMNIC window (Figure 2.10), go to the Atlμs menu and select 'Show Atlμs Window….' This will launch the program and automatically scale the main OMNIC window to allow viewing and operating both programs simultaneously. The main Atlμs window is shown in Figure 2.14. The image on the right is the live image coming from the microscope (if you don't see anything, make sure the microscope is in View mode, and that the video camera power supply is on (Figure 2.4).

The first thing to make sure is that the correct calibration is open. In the title bar of the screen shown in Figure 2.14, it states that the [32x objective calibration] is open. If you are using a different objective with the microscope, go to the Image menu, and select 'Open Calibration'. There are calibration files for the 10x, 15x, and 32x objectives; select the correct one for what you are using. Now the scale on the video image will be correct and you can correctly set up mapping experiments.

The easiest way to begin setting up a mapping experiment is to use one of the blue colored tools at the bottom of the Atlμs window. They are, from left to right, pointer to select and move objects, a line map drawing tool, an area map drawing tool, a points map drawing tool, a background point selector, a target to move the x-y stage to a specific position, drawing a circular aperture, drawing a rectangular aperture, a ruler, and a way to add Text to the image. Most people want to do line or area maps, so select the line or

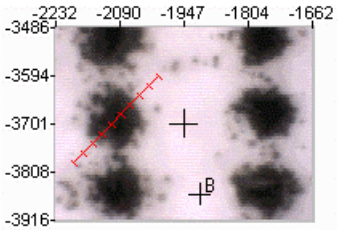
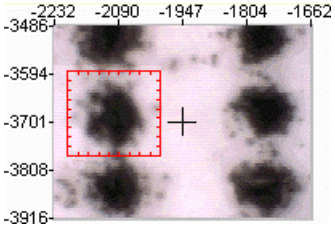


Figure 2.15. Examples of drawing a line map (left) and an area map (right) on the live microscope image (red lines with step marks indicated).



area map drawing tool and draw on the microscope image where you would like the software to acquire an IR map of your sample. To see and draw on the microscope image more clearly, expand the image by going to the Image menu, and select a larger image size up to 640×480.

Now that you have your map coordinates roughly drawn onto the microscope image, open the Collect menu, and select Map Setup. This will open a window where you can specify the details of the map you want to collect. The first tab, Dimensions, will load the coordinates for the map you drew from the previous paragraph. You can edit these coordinates, change the step size, and see an estimated amount of time for acquiring the map. If you make changes here, select Apply to update the drawn map on the microscope image as well as the time estimate.

The next tab is labeled Aperture and allows you to specify that you are using an aperture. Since the synchrotron beam is very well focussed, most users to not use an additional aperture so None is usually selected.

The Collect tab allows you to give your map a descriptive title (not the file name, that will come after the map has been acquired), designate the number of scans to be co-added at each mapping point, the resolution for the scans, the apodization to use for the Fourier processing, and the Final Format to save the data in. The apodization is usually left at Happ-Genzel, and we strongly recommend that you select the Final Format to be Single Beam. Once a map file has been processed including dividing by a background spectrum, you cannot reprocess the map again. By saving the map in Single Beam format, you will need to manually process the map with a background file, but it will allow you to reprocess the map as many times as you need to for getting your data correct. If there was a problem with the background file, or you decide to acquire a new background spectrum, you can still reprocess your map file.

The Options tab lets you select how the video frame is saved, and also if you want to manually help focus during the mapping experiment. Typically 'Save Video Frames In Map File', 'Store Map With Relative Coordinates', 'Prompt Before Collecting Data', and 'Collect Single Background For Entire Map' are selected. Only select 'Auto-Pause Before Each Spectrum' if you want the program to pause data collection at every map point to allow you to refocus or otherwise check on your samples (this takes a long time and you have to click OK over and over so choose this only if you really mean it!).

The Focus tab allows you to use the Autofocus capabilities of the microscope. The autofocus system only uses the visual video image for focussing and not the IR signal, so until we have developed software to improve this feature, we suggest you leave 'Do Not Focus During Map Collection' selected.

Now that you have completed the Map Setup, simply go to the Collect Menu and select Collect Map. If you have 'Prompt Before Collecting Data' selected, you will now see a prompt asking if it is OK to capture the video image. If you are visually in focus, select OK. Now you will get a prompt to let you tell the software that you are ready to collect

the map. DO NOT SELECT OK YET. First move this prompt window up and out of the way so that you can get to the Collect Menu in the Main OMNIC window. Select Experiment Setup, and the Bench tab, then adjust the fine focus on the microscope to get the maximum IR signal in the interferogram. Close this window by clicking OK, and then go back to the Atlμs prompt and select OK to let the map begin acquisition. When the map has completed, you will be prompted to enter a file name to save the map file into your Maps directory.

Once the Map data is saved, there is one more step to do - reprocess the data with an appropriate background spectrum. To do this, first save the map again with a new name by going to the Atlμs menu and selecting 'Save Map As….' Give is a different name; most users add a %R or Abs at the end of the name to indicate the final format that you are about to process the map into. Once it's saved, go to the Atlμs menu, and select Reprocess Map. Say OK to the warning that you will overwrite your data (that is why you just saved the data with a new filename). You will now see a new dialog box where you can select the final format you want, and you can Browse for a background spectrum to use in the processing. Click OK when you are done and the map will be reprocessed.

When you are working with a **Line Map**, you can view the data in two formats, Waterfall and Contour Map. Examples of the same data in the two formats are shown in Figures 2.16 and 2.17. The Waterfall shows you each spectrum with the axis going back away from you being distance in microns. The Contour Map is like looking straight down on the Waterfall; the vertical axis is position in microns, the horizontal axis is wavenumbers, and the colors represent intensity (colors can by set in the Atlμs / Display Options menu).

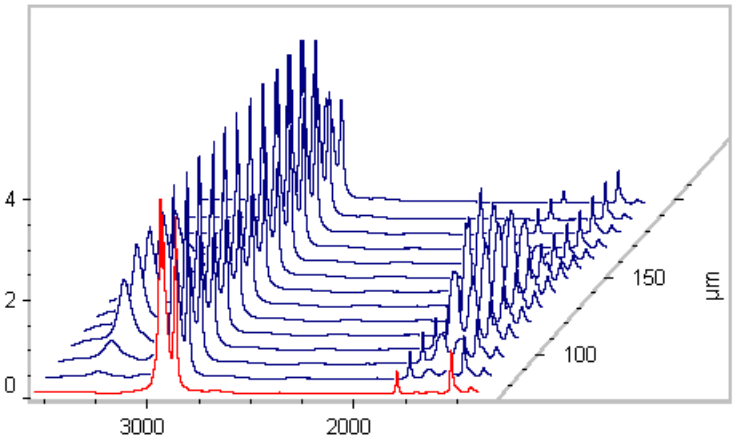


Figure 2.16. Example of line map data shown in Waterfall format. The active spectrum is shown in red.

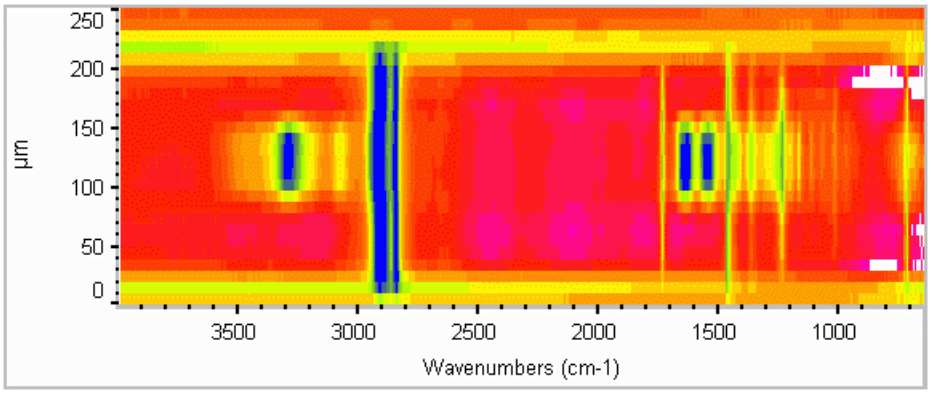


Figure 2.17. Example of line map data shown in Contour Map format. You can use the Spectral Selection Tool **to select an active spectrum from within the Contour Map.**

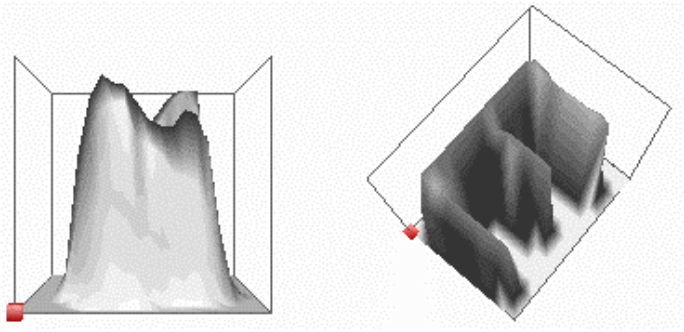


Figure 2.18. Example of a 3D display for an area map. The second example has been rotated to show the "shape" of the data more clearly.

To extract the intensity or area of a specific peak as a function of position, select the Profile Setup button at the bottom middle of the Line Map display window. You will be asked to enter the appropriate wavenumbers, then select Create. A new window will open with your profile peak as a function of position along the line map.

When you are working with an **Area Map**, the window will be split into several active areas. The top panel will show the current active spectrum, and the video image with the point of the active spectrum marked. The lower left panel shows a contour profile map with the profile shown with the colors representing the intensity of the peak, selected in the Profile Setup and Display Setup, as a function of position. The lower right panel shows a 3D representation of the profile map that can be rotated for a better view using the mouse, as shown in Figure 2.18. Just like in Line Maps, you can use the Spectral Selection Tool to select a specific spectrum from the Area Map's contour profile map, or the video image. The selected active spectrum's position is marked with a red cube in the 3D representation.

The Atlμs help files can give you a lot more information about the program and other ways to process and view map files.

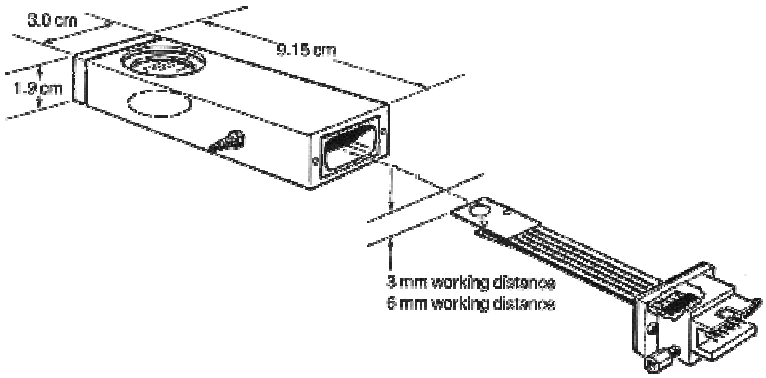


Figure 2.19. Drawing of the MMR cryostat system.

MMR Cryostat Sample Holder

We have two MMR micro-miniature refrigerators for measuring samples at temperatures from 70 to 540K or 70 to 730K in the IR microscope. The system consists of a cryogenic refrigerator that derives its cooling power from the Joule-Thomson expansion of a high-pressure gas (usually Nitrogen or Argon) sealed inside a vacuum shroud with IR windows as drawn in Figure 2.19. The sample is mounted on the copper tab attached to the end of the refrigerator using the 6mm working distance to allow for the focal depth of the IR microscope's objective. Once the refrigerator is sealed inside the vacuum shroud, the unit is placed in a custom aluminum holder (pictured in Figure 2.20) which then fits in the microscope x-y stage.

To use these refrigerators for the first time, please have Mike help walk you through it once. This manual will give you some general procedures to follow, but please do not try to operate the refrigerators with only this manual.

Once you have a sample mounted on the copper sample clip, insert the refrigerator into the vacuum chamber with the rubber interface gasket in place. Tighten the two small screws to get a good seal on the gasket. Place the unit on the microscope stage using the specially made aluminum holder. Attach the high-pressure gas lines from the gas cylinder (high purity Nitrogen or Argon) to the long tubular filter, then to the refrigerator inlet. Attach the small rubber hose to the gas outlet and to the flowmeter. Connect the temperature controller electronics ribbon cable to the refrigerator making sure the tab is on the correct side - the connector should slide on easily. Finally attach the vacuum hosing to the pump out port on the vacuum chamber.

Now that the system is fully assembled, follow the steps below to get your sample to the proper temperature:

1. Run about 500-psi of Nitrogen (or Argon) through the system for about 30 seconds to purge it of any moisture that may have collected during nonuse.
2. Turn on the vacuum pump and wait for the pressure to reach ~10 millitorr.

3. **For cooling:** turn up the high-pressure nitrogen regulator to 1800-psi MAXIMUM. Make sure the flowmeter shows that gas is flowing - a typical reading is 3.5. The sample should now begin to cool. **For heating:** skip this step.
4. Turn on the K-20 temperature controller. Check that the temperature reading is reasonable, then set your desired temperature set-point (see below for how to operate the K-20).
5. Once the temperature has stabilized, conduct your experiment.
6. After the experiment is finished, turn off the high-pressure gas. Allow the refrigerator return to near room temperature, then turn off the vacuum. You can now power down the K-20, disconnect the various connections, and remove the MMR system from the microscope and return everything to where they came from.

Operation of the K-20 temperature controller.

All functions of the K-20 controller can be accessed via the blue C-2000 keypad and display. A summary of the most common commands for operating the unit is shown below. More details can be found in the MMR manuals, but most users can get by with just the following.

Command	How to remember	What you get	Typical response
TE	TEmperature	Displays current temperature of the sample stage.	`0243.45 K`
WK	What Kelvin?	Displays current set-point temperature.	`0130.00 K`
SK ###.##	Set Kelvin	Set's the set-point temperature to the value you enter for ###.##.	`OK`
PO	POwer	Displays current heater power level.	`01.023 W`

MOx	MOnitor every x seconds	Displays the Temperature and Power every x seconds, where x is 1-9. This will continue until you tell it to stop by pressing any key.	`OK AUTO>`
			
			`274.21 K`
			`00.000W AUTO>`
			
			repeats …