USER MANUALS

Thermo Nicplan

Measurements at the ALS

If you are a new User, please **WAIT** for the beamline scientists to arrive. If you experience problems with the instrument, please contact the beamline scientists for assistance.

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Users may optimize the microscope endstation and fill the detector with lN$_2$ after receiving training from the beamline scientists.

The beamline alignment should be optimized by staff only.

Switching Between Modes The StarTech box shown in Fig 1 toggles between computer 1 (measurement computer) and computer 2 (old bench computer). Use the computer 2 Omnic program to change measurement modes by navigating to *Collect → Experiment Setup → Bench Tab*. The drop-down arrow indicated by the red boxed section changes the measurement mode between Reflection and Transmission as in Fig 2. Click *OK* to close the bench window. Switch back to computer 1 and proceed with the measurement. The *Infrared* indicator on the microscope (red box in Fig 3) should light up with the selected operation mode. Press the *View* button to enable viewing by eye or CCD camera.

The IRdata Network Please save all data to your personal folder on the IRdata network drive. This will enable access from any of the IR group computers. Any data saved directly to the measurement computer may be deleted during periodic maintenance. The beamline scientists will work to preserve data on the IRdata network, but Users should retain a complete copy of their data in the event of network failures.
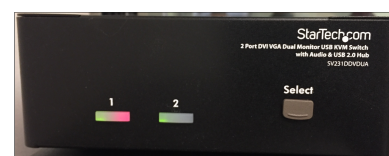

Figure 1: The StarTech box is used to toggle between computer for changing the measurement mode: i.e. between transmission and reflection measurements.
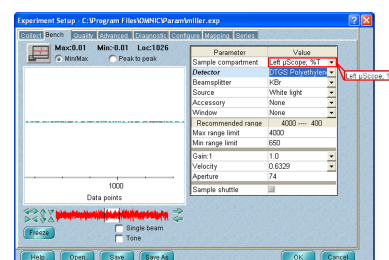

Figure 2: Image of the *Bench* tab on computer 2. Use the drop-down menu indicated by the red box to change between %R and %T operational modes.

Figure 3: When the measurement mode changes, the appropriate green LED will illuminate on the microscope. In this image, the system is configured for viewing the sample in Reflection.

Spectromicroscopy with the Thermo Nicplan

Preparing the Detector Wearing safety glasses, fill the small Nicolet thermos with lN$_2$. The MCT has a *shallow* funnel built-in to the microscope (see Fig 4); take care to pour small amounts of lN$_2$ very slowly until the funnel is cold. One thermos of lN$_2$ fills the detector and lasts for 8-10 hours.

Note: It is recommended that Users keep a log of the lN$_2$ fill times. Do not put more than one thermos of lN$_2$ into the detector within the 8-10 hour period as overfilling can damage the microscope

Changing File Path Options To change the file path for saving data, go to the *Edit* menu and select *Options*. Specify the initial and auto save file path to your personal folder on the network as shown in the red boxes of Fig 5.

In the *Collect* tab of the *Experiment Setup* window, verify your specified auto save path is correct as in Fig 6. Add an experimental title and a base name. The software will increment by 0001 if the base name is not changed between measurements.

Navigating Omnic The Omnic program uses a series of windows for acquiring and processing data: a measurement acquisition window, windows for completed spectra (Users have the option of adding subsequent measurements to an existing completed spectra window via a pop-up box once collection of a spectrum is completed), and the Atlμs window for visible viewing with a camera (useful for spectral mapping). The acquisition window will open automatically once data collection has started. Users can manipulate the acquired spectra and also save data to different formats within the completed spectra window. In the Atlμs window (17), the sample stage can be moved to the cursor location using the tool (**which one?**) and the ruler tool can be used to measure distances in μm. However, the objective magnification and Atlμs magnification need to be manually set to the same value or the distances displayed in the Atlμs window will be incorrect. The *Mosaic* command will create a large image of tiled scans if the area of interest is large.

Saving Spectral Data Omnic automatically outputs data in a proprietary file type .SPA and will save data to the directory specified *Collect* tab (red boxed file path in Fig 6). Verify the options *Save Automatically* and *Save Interferograms* are both selected as this will enable the reprocessing of data to different spectral resolution or data formats, if needed. To save data as .CSV or .TXT, select spectra by clicking on them individually in the completed spectra window. Then, go to *File → Save As* to change the file format. See the section on configuring spectral mapping for instructions on saving map data.
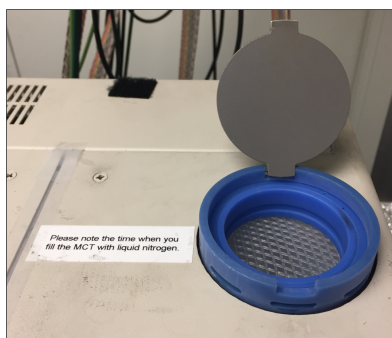

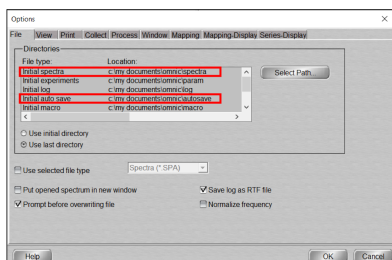Figure 4: The microscope funnel is built-in to the housing. Be careful not to overfill.

Figure 5: File tab in the Options window. The red boxes indicate suggested file paths to change. Select the file type and then click the Select Path button to specify the desired file path.
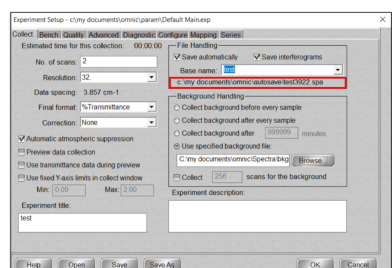

Figure 6: Collect tab in the Experimental Setup window. The red box indicates the present file path.
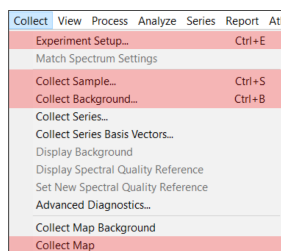

Figure 7: The *Collect* menu in Omnic. The most frequently used selections are highlighted in red. *Experiment Setup* is where configurations of the measurement can be altered.

Transmission Measurements

Aligning the Condenser in the Visible and Infrared Insert the aperture labeled "Lower" and align the visible light in *View* mode using the condenser micrometer knobs on the left side of the microscope, below the sample stage indicated by the arrows in Fig. 8. The position is correct when the sharp edges of the "Lower" aperture are in focus. Once the "Lower" aperture is in sharp focus, remove the "Lower" aperture and turn off *View* mode.

Next, optimize IR beam by maximizing the peak to peak FTIR signal. In the menu bar, go to *Collect → Experiment Setup* and open the *Bench* tab as shown in Fig. 9. Fine-adjust the condenser micrometer knobs to maximize the peak to peak signal: start with the x and y directions and optimize before moving on to the focus knob. Iterate between all three directions until the maximum signal is achieved. A typical peak to peak value is around 6 for a gain setting of 1 in transmission with the 32x objective and the lower aperture removed. It is normal for the optimized IR position to be slightly different from the visible.

Note: Changing the "Optical Velocity" shifts the noise frequency components: faster scanning moves noise to lower frequencies. The "Aperture" setting in Omnic doesn't alter the synchrotron beam.

Collecting Transmission Spectra In the *Collect* tab of the *Experiment Setup* window shown in Fig. 10, specify the number of scans, resolution, format, and file extension. Use a quick scan to verify the settings are appropriate. Increasing the number of scans and/or the resolution increases the acquisition time; an estimate of which is displayed above the scan settings.

Note: Spectra with a resolution finer than 4 cm^{-1} may experience noise in the form of interference effects. The actual measurement time is approximately a factor of 1.5x longer than the calculated value shown in the Mapping tab.

Background Measurements Depending on the individual measurement plan, Users may collect background spectra on a reference sample or clean substrate. For single spectra, go to *Collect → Collect Background* as indicated in the red highlighted area of Fig. 7. To designate a specific background file, select *Use Specified Background File* under *Background Handling* in the *Collect* tab of the *Experiment Setup* window. This will direct the software to the appropriate .SPA file for % Transmittance calculations. Alternatively, to collect spectra without the software applying a reference, select the option highlighted in red in Fig.10 to *Collect spectra after (999999) minutes*.

With a substrate or reference sample in place, the condenser alignment may need to be adjusted slightly, starting with the focus (red knob). The x and y directions may only require a small change, if at all.
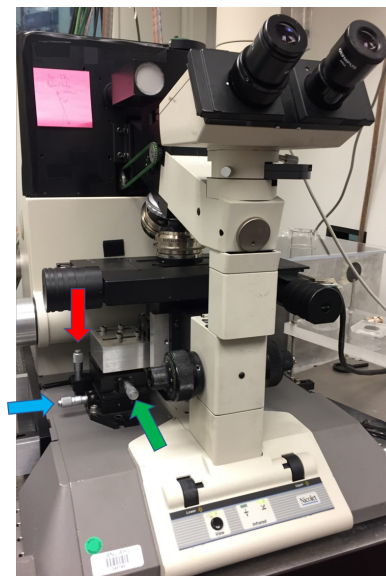

Figure 8: The micrometer stage for the condenser is shown. The red arrow indicates the micrometer knob used to focus. The blue and green arrows indicate the x-y positioning micrometers of the condenser, respectively.
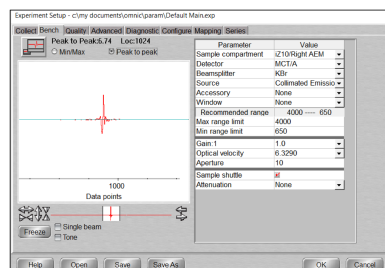

Figure 9: Bench tab of the Experiment Setup window. The FTIR centerburst is displayed in red. The peak to peak signal should be monitored while adjusting the microscope alignment.
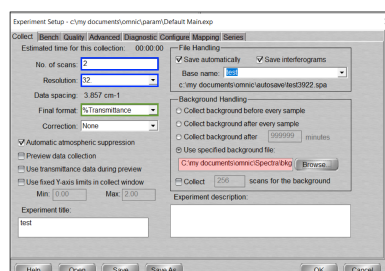

Figure 10: *Collect* tab of the *Experimental Setup* window. The scan settings are boxed in blue and the measurement mode is boxed in green. The background file path is highlighted in red.

Reflection Measurements

Verify the microscope is in Reflection mode as indicated by the green LED on the base of the microscope (see Fig. 3). If not, follow the instructions for *Switching Between Modes*.

Optimizing the Visible and Infrared In *View* mode, use the knobs on either side of the microscope upright support to focus the visible light on a gold sample.

Note: If the focus is off significantly or the gold sample is very clean, the upper aperture can be used as a reference point - raise or lower the stage until a dark rectangular box appears in the eyepiece. The sample surface is at essentially the same position.

Aligning the Visible and Infrared Optimize IR beam by maximizing the peak to peak FTIR signal. In the menu bar, go to *Collect → Experiment Setup* and open the *Bench* tab as shown in Fig. 9. Fine-adjust the focus knobs on either side of the sample stage to maximize the peak to peak signals. A typical peak to peak value on gold is **VALUE?** with a gain of 1 and the 32x objective. The optimized IR position and the visible focal plane should be fairly close.

Collecting Reflectance Spectra In the *Collect* tab of the *Experiment Setup* window shown in Fig. 10, specify the number of scans, resolution, format, and file extension. Use a quick scan to verify the settings are appropriate. Increasing the number of scans and/or the resolution increases the acquisition time; an estimate of which is displayed above the scan settings.

Note: Spectra with a resolution finer than 4 cm^{-1} may experience unwanted noise in the form of interference effects. The actual measurement time is approximately a factor of 1.5x longer than the calculated value shown in the Mapping tab.

Background Measurements Depending on the individual measurement plan, Users may collect background spectra on gold, a reference sample, or a clean substrate. For single spectra, go to *Collect → Collect Background* as indicated in the red highlighted area of Fig. 7. To designate a specific background file, select *Use Specified Background File* under *Background Handling* in the *Collect* tab of the *Experiment Setup* window. This will direct the software to the appropriate .SPA file for % Reflectance calculations. Alternatively, to collect spectra without the software applying a reference, select the option highlighted in red in Fig. 10 to *Collect spectra after (999999) minutes*.

Configuring Spectral Mapping

Align the microscope for transmission or reflection measurements, as detailed above. For transmission (reflection) mapping, place the 5 μm pinhole (gold reference) on the sample stage. Then, center and focus on the pinhole (defect on the gold reference) with visible light. Open the Atlμs window and use the area select tool (indicated by arrow and red box in Fig. 11) to draw a box around the pinhole (gold defect).

Alignment Mapping Configure a quick mapping for aligning visible to the IR as follows: in the *Experiment Setup* window *Collect* tab, change number of scans to 2, Resolution to 32, Format to Single Beam, and turn off automatic atmospheric suppression as shown in Fig. 12.

Note: If the format is not single beam, a background error will appear when attempting to collect the map.

In the *Mapping* tab shown in Fig. 13, set the step size to 2 μm and click *Apply*. Also select the *Advanced Mapping Options* in the red box to open the dialog and check *Save video frames in map file* and *Prompt before collecting data* in Fig. 14. This will enable reminders during data collection for turning the *View* button on and off.

After configuring the mapping options, verify that the pinhole (gold defect) area is still selected (i.e. the blue box with dots in Fig. 15). Re-select the area if necessary. Go to *Collect* and then *Collect Map* to start collecting the alignment map (Fig. 15). If the settings are correct, Users will be prompted to verify the microscope is in video (*View*) mode. Toggle the microscope *View* button as necessary. The mapping program will then collect an optical picture of the scan area. A second prompt should then pop up to verify the system is ready for IR acquisition. Make sure the *View* button is turned off (manually switch to IR mode) before clicking *OK* on the prompt. The flip mirror associated with switching between the *View* and IR measurement modes occasionally sticks and the *View* button may need to be pushed a second time. Verify the microscope is in IR measurement mode before starting the IR acquisition.

Once the quick map of the pinhole (gold defect) is complete, the map window will appear similar to what is shown in Fig. 16 with the IR map in the upper left and the visible map in the upper right. In transmission, the IR light is circular; while in reflection mode, the beam is a diagonal ellipse. The blue x and y profile curves help identify the center of the IR hot spot relative to the sample stage position. The cursor in the red single beam spectrum located in the center of the window can be used to locate the peak signal of a particular frequency of interest, if needed, so that visible alignment can take place relative to the focal spot of that frequency.
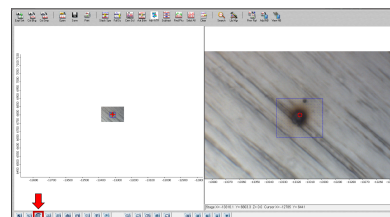

Figure 11: The area select tool is indicated in the Atlμs window by the red arrow and box. Use the tool to select an area around the pinhole to check the alignment of the visible and IR light.
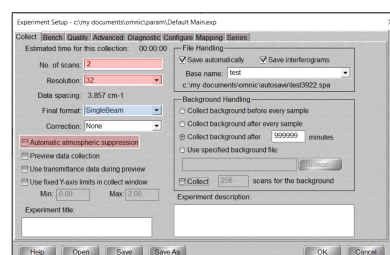

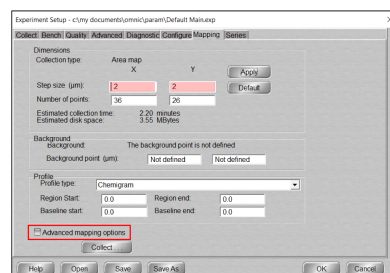Figure 12: Recommended settings for a quick alignment map.

Figure 13: *Mapping* tab of the *Experiment Setup* window. The measurement spacing needs to be smaller than the feature of interest - in this case, the 5 μm pinhole. Click *Apply* after changing the step size. Check *Advanced Mapping Options* to enable reminder prompts.
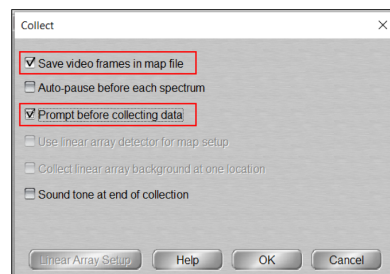

Figure 14: *Advanced Mapping Options* prompt. Select the options boxed in red to enable reminder prompts for data collection.
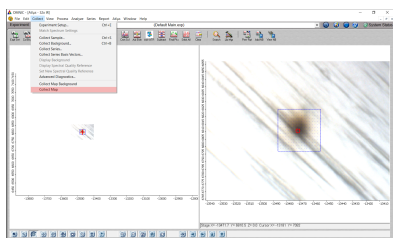
Figure 15: Atl*μ*s window with pinhole area selected. *Collect Map* command is highlighted in red. This will start the data acquisition.
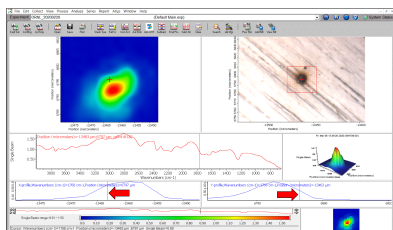

Figure 16: Quick spectral map of the pinhole to verify alignment of the visible and IR light.
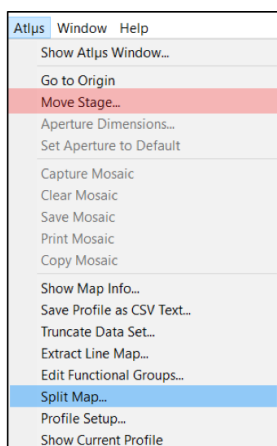

Figure 17: *Atlμs* Options. The *Move Stage* command is highlighted in red and the *Split Map* command is highlighted in blue.

Note: The IR beam expands with wavelength; use the red frequency slider to see the focus in the frequency range of interest and verify it is aligned with visible.

Keep in mind that the resolution of the IR light (2-10 μm) is diffraction-limited, so attempting to overlap the visible and IR much below 2 μm isn't practical. If the visible and IR are misaligned by more than 2 μm, please contact the beamline scientists to have it adjusted.

Collecting Mapping Data Once the visible image is aligned to the IR, data collection can proceed following a similar process as what was used for alignment. Select an area of interest using the area select tool. Enter the spectral configuration requirements in the *Collect* tab of the *Experiment Setup* window in Fig. 10. Verify an appropriate background file is selected and that % Transmittance (Reflectance) is selected as the *Final Format*.

In the *Mapping* tab, enter the step size and click *Apply*, making sure to select *Advanced Mapping Options* and enabling the prompts shown in Fig. 13. The actual measurement time is approximately a factor of 1.5x longer than the calculated value shown in the Mapping tab.

Once completed, the spectral map data will be shown in a window similar to Fig. 16. Data can then be manipulated within the Atl*μ*s window.

Note: Switching from the alignment pinhole (gold defect) to a reference sample, or switching between samples of different thicknesses will likely require re-focusing of the condenser (stage) to maximize the IR signal transmitted through (reflected from) the material. Use the Bench tab to monitor the live IR signal for optimization.

Exporting Spectral Maps For Users without access to a version of Omnic with mapping features, the data can be split into individual spectra by selecting Atl*μ*s and then clicking *Split Map* (shown highlighted in blue in Fig. 17). The data can then be batch converted into another file format by navigating to the appropriate file location, selecting all of the map spectra, opening, and converting to .CSV.