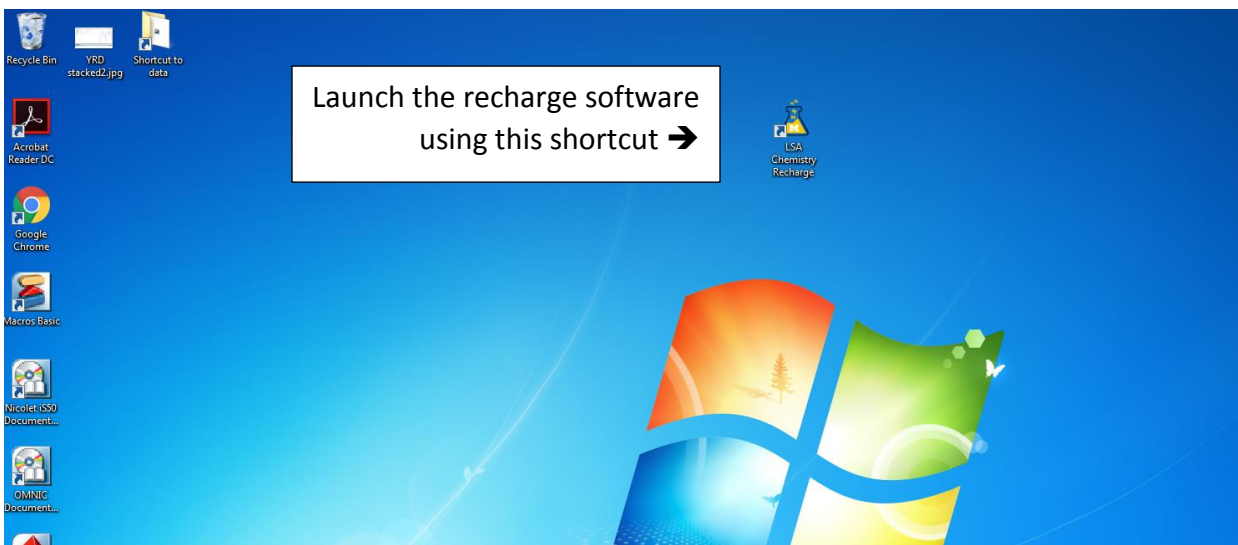


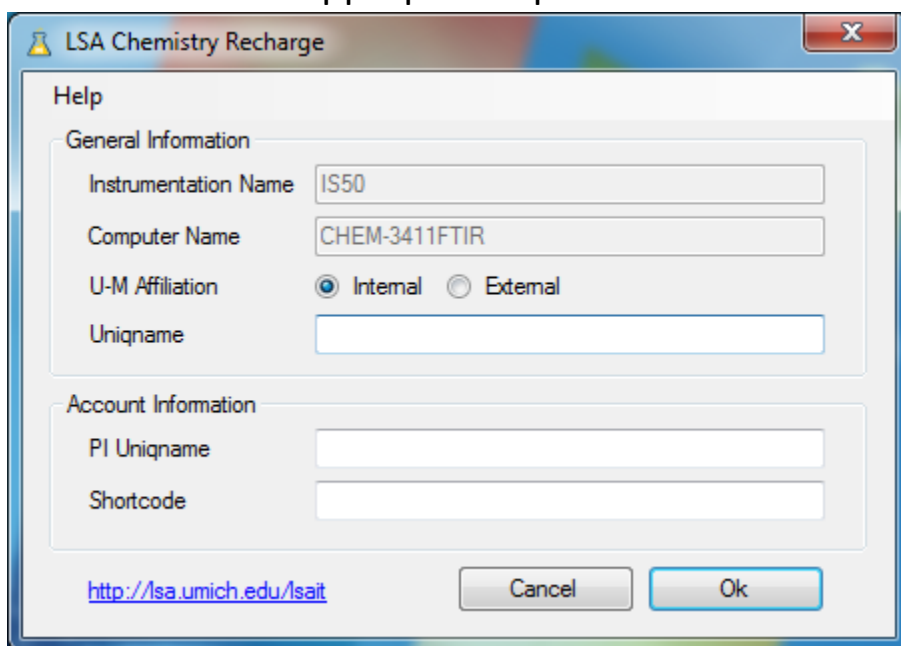
Nicolet iS50 FTIR User's Booklet

I. Getting started

- 1) Start your session by launching the LSA Chemistry Recharge software. This software keeps track of the amount of time that you are using the instrument.



- 2) Type in your unique name, your PI's unique name and your shortcode in the appropriate spaces. Then click the OK button.



The image shows a Windows-style dialog box titled "LSA Chemistry Recharge". It has a "Help" icon in the top-left corner and a close button (X) in the top-right corner. The dialog is divided into two main sections: "General Information" and "Account Information".

General Information:

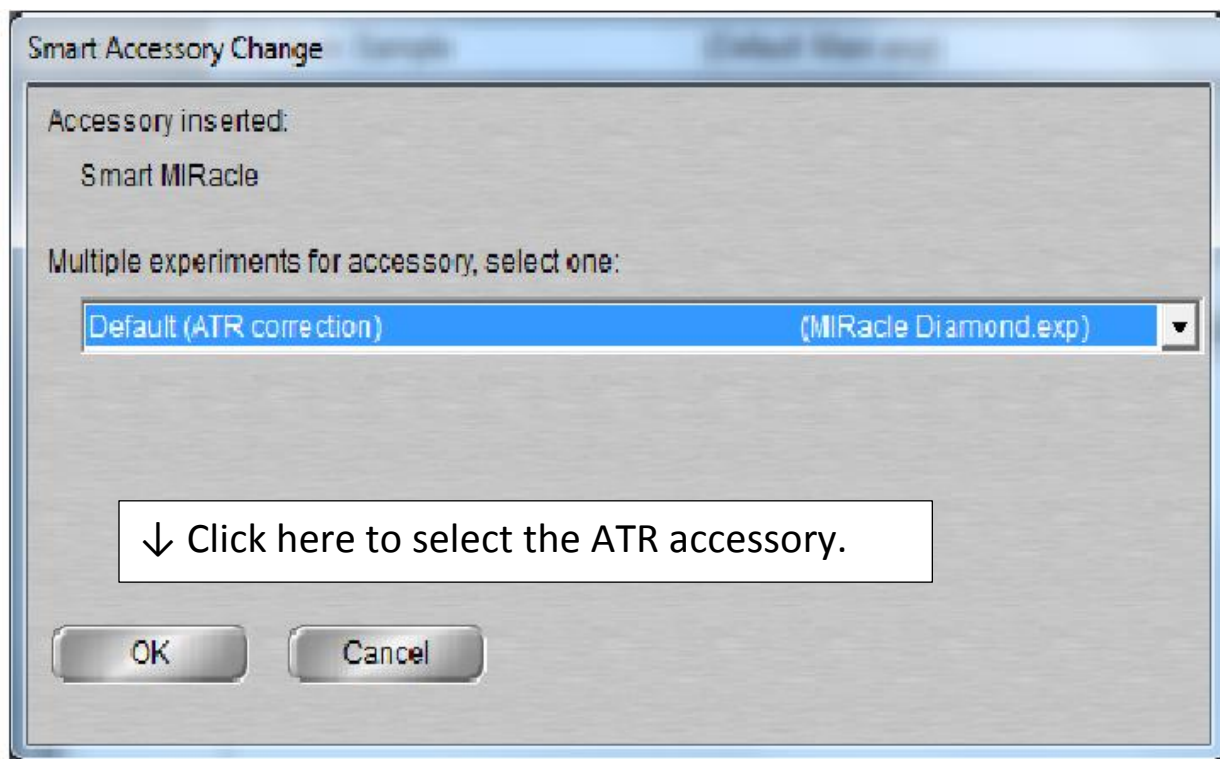
- Instrumentation Name: Text box containing "IS50".
- Computer Name: Text box containing "CHEM-3411FTIR".
- U-M Affiliation: Radio buttons for "Internal" (selected) and "External".
- Uniqname: Empty text box.

Account Information:

- PI Uniqname: Empty text box.
- Shortcode: Empty text box.

At the bottom left is a URL: <http://lsa.umich.edu/lsait>. At the bottom right are "Cancel" and "Ok" buttons.

- 3) This will launch the Omnic software used to control the FTIR instrument.
- 4) Click on OK to select the default ATR setup.



The image shows a Windows-style dialog box titled "Smart Accessory Change". It has a "Smart" button and a "Cancel" button in the top-left corner. The dialog contains the following information:

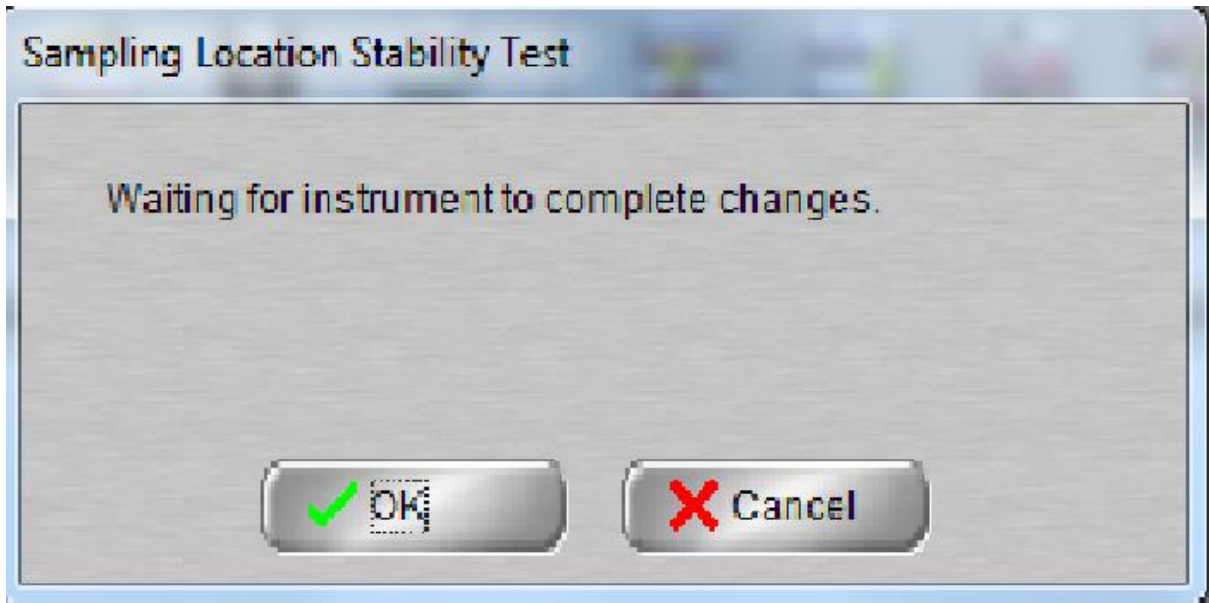
Accessory inserted:
Smart MIRacle

Multiple experiments for accessory, select one:
A list box shows "Default (ATR correction)" selected, with "(MIRacle Diamond.exp)" to its right and a dropdown arrow.

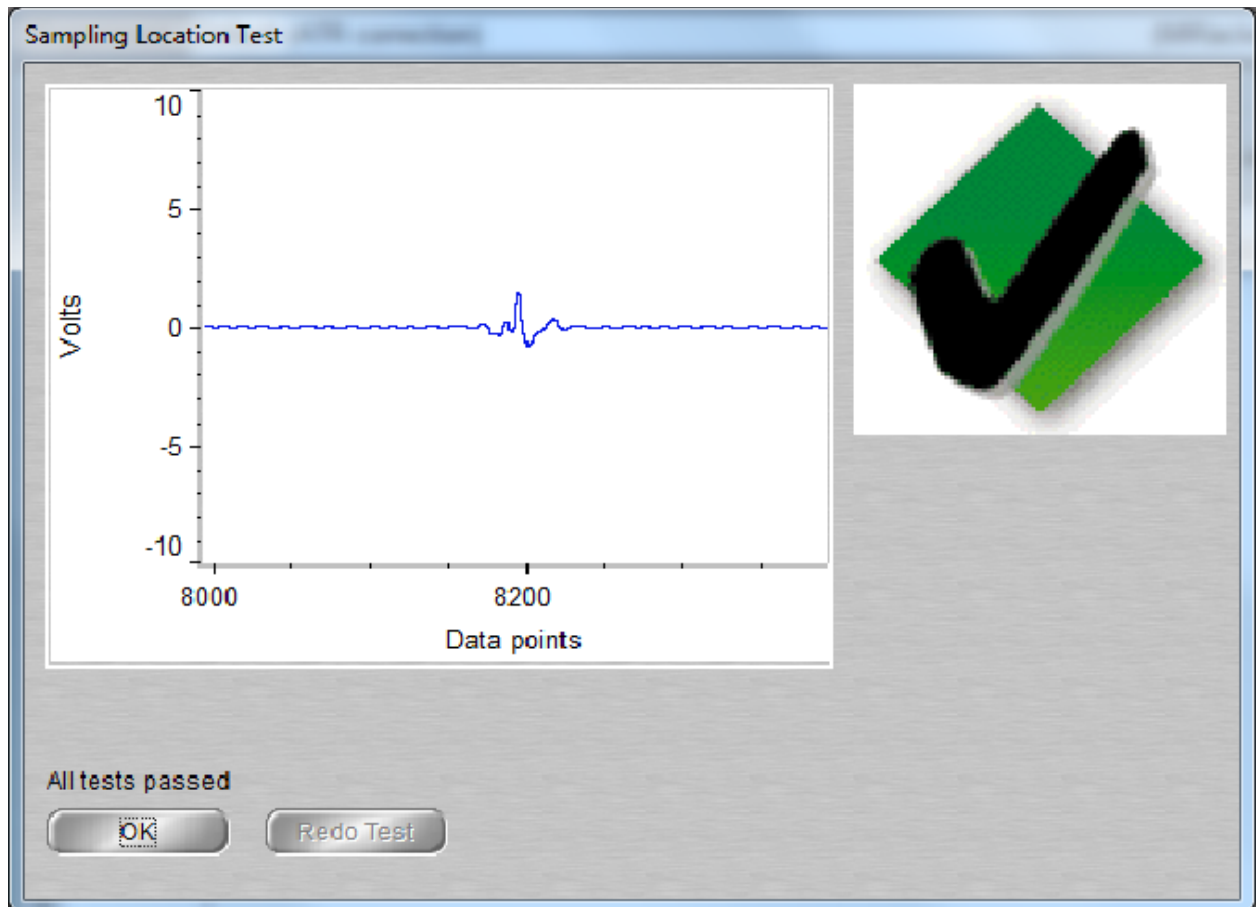
Below the list box is a text box containing the instruction: "↓ Click here to select the ATR accessory."

At the bottom are "OK" and "Cancel" buttons.

5) Click OK, or wait until the instrument completes the changes.



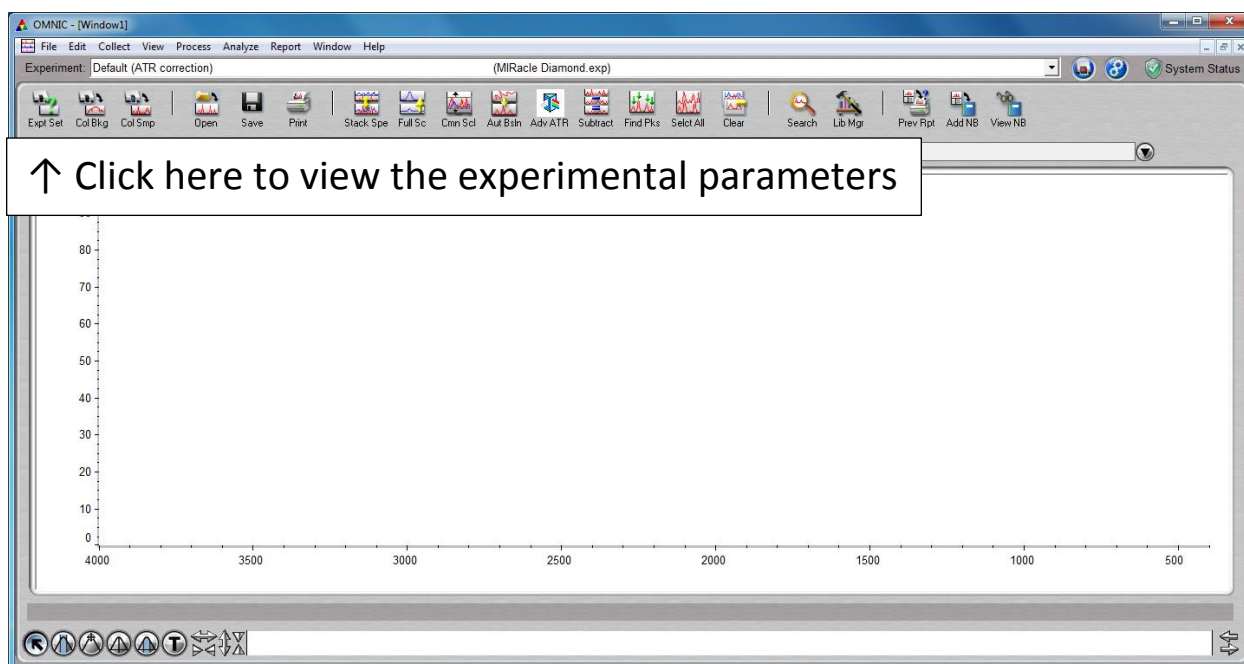
6) Click OK once the location test is complete.



7) The instrument is now ready to begin analysis. The experimental parameters can be checked, or modified. Then, a background spectrum is acquired, followed by acquisition of one or more sample spectra. One background spectrum is sufficient for numerous sample spectra, as long as the experimental parameters stay the same.

II. Selecting experimental parameters:

- 1) Click on the Experimental Setup button in the upper left corner of the screen.



- 2) The default parameters for a standard characterization spectrum are 8 scans and a resolution of 4. The number of scans can be chosen by entering the desired value. Resolution values are chosen from the drop down menu.

Experiment Setup - c:\my documents\omnic\param\MIRacle Diamond.exp

Collect Bench Quality Advanced Diagnostic **Configure**

Estimated time for this collection: 00:00:11

No. of scans:

Resolution:

Data spacing: 0.482 cm⁻¹

Final format:

Correction:

☐ Automatic atmospheric suppression

☐ Preview data collection

☐ Use transmittance data during preview

☐ Use fixed Y-axis limits in collect window

Min: Max:

Experiment title:

File Handling

☐ Save automatically ☒ Save interferograms

Base name:

Background Handling

☐ Collect background before every sample

☐ Collect background after every sample

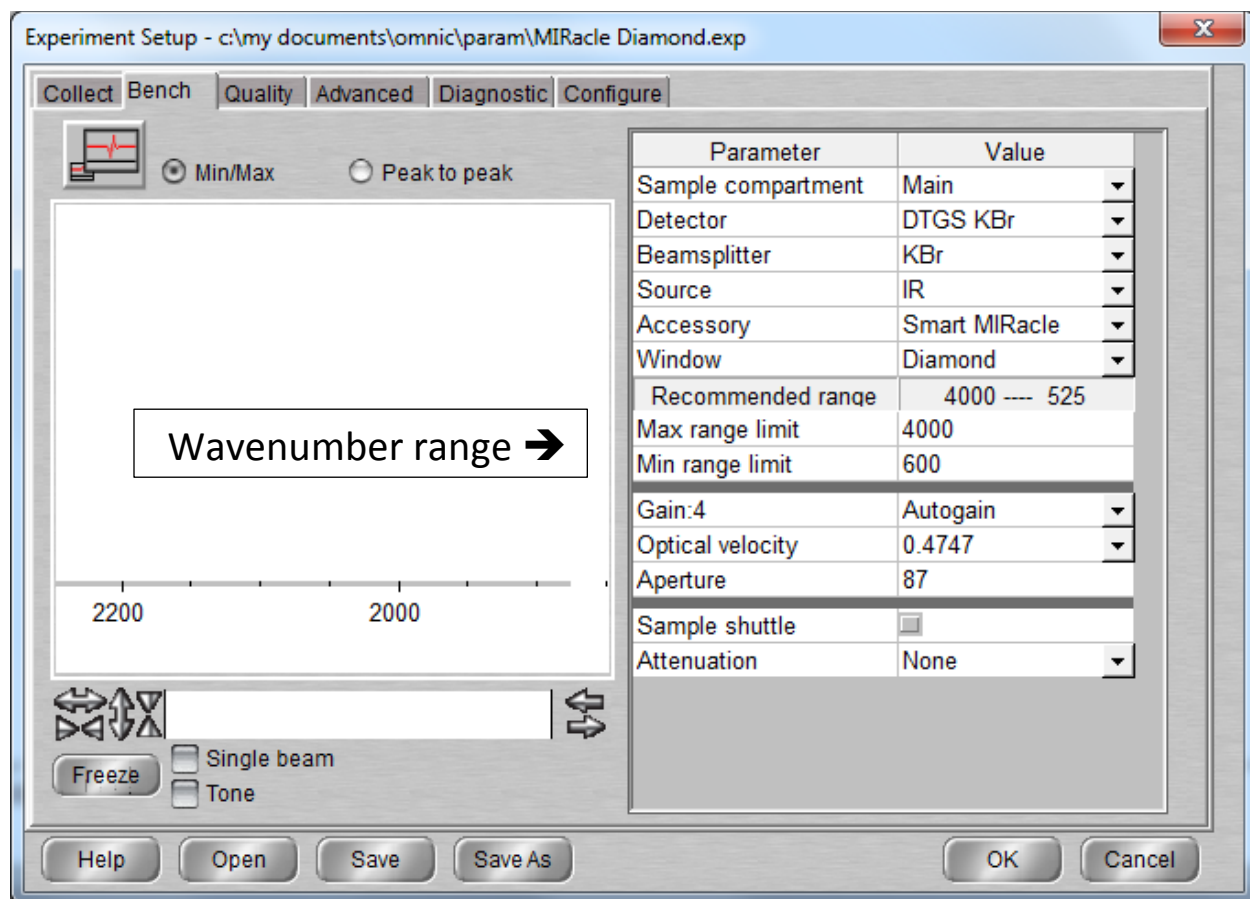
☒ Collect background after minutes

☐ Use specified background file:

☐ Collect scans for the background

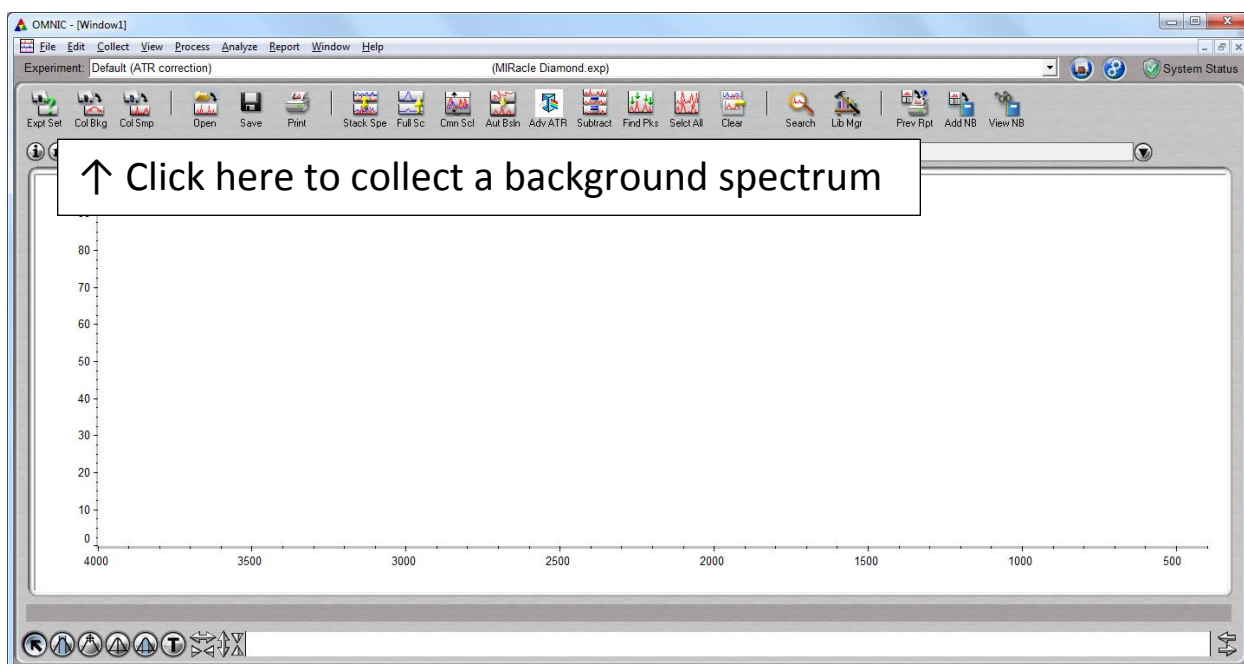
Experiment description:

- 3) Click on the Bench tab to view the range of wavenumbers covered by the scan. These values can be change as necessary.

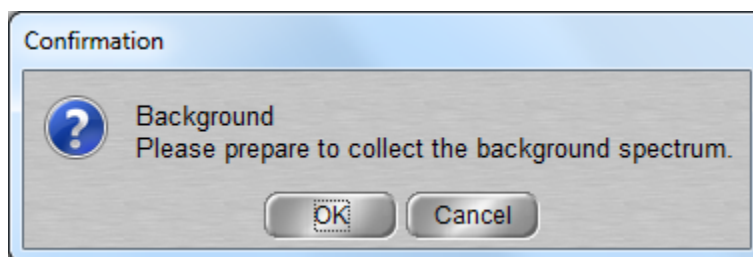


III. Collecting a background spectrum:

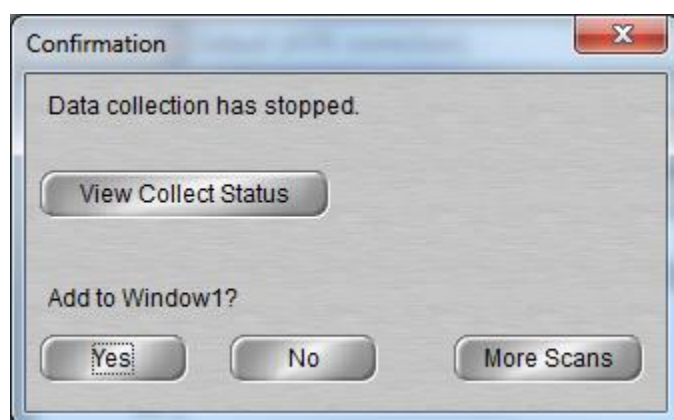
1. Click on the Collect Background button in the upper left corner of the screen to collect a background spectrum.



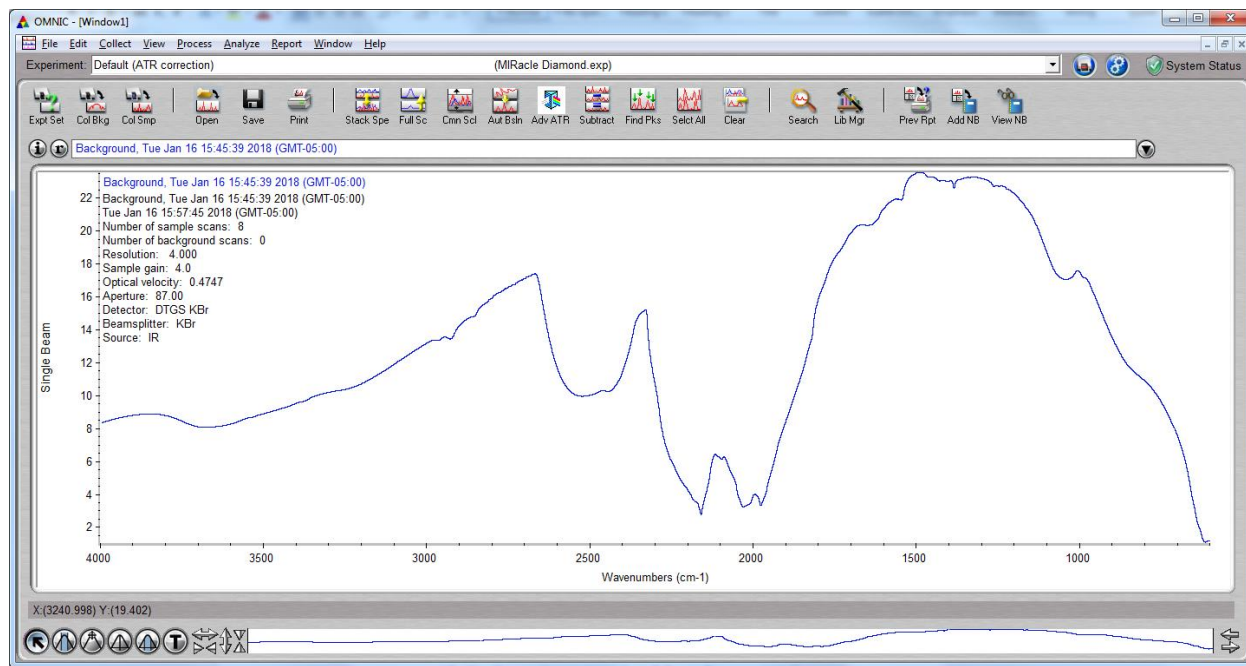
2. You will then be prompted to confirm that you want to collect a background spectrum. If you haven't yet cleaned the ATR crystal, after the previous user, now would be a good time. Click "OK" when ready.



3. After confirmation, the instrument begins to collect data. The steps that the instrument takes can be viewed in the bottom left corner of the screen. First, a couple of setup processes are completed, then a countdown of the chosen number of scans begins (1 of 8, 2 or 8, etc.). When the collection is complete, the spectrum is displayed and you are prompted to save the data to the currently opened window. If the data are acceptable, click “Yes” to save the spectrum.

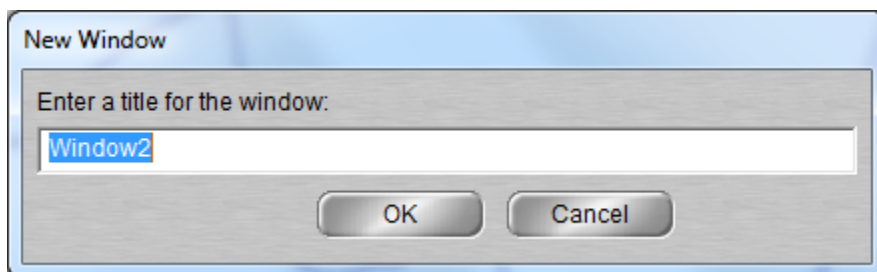


4. Below is a typical background spectrum.

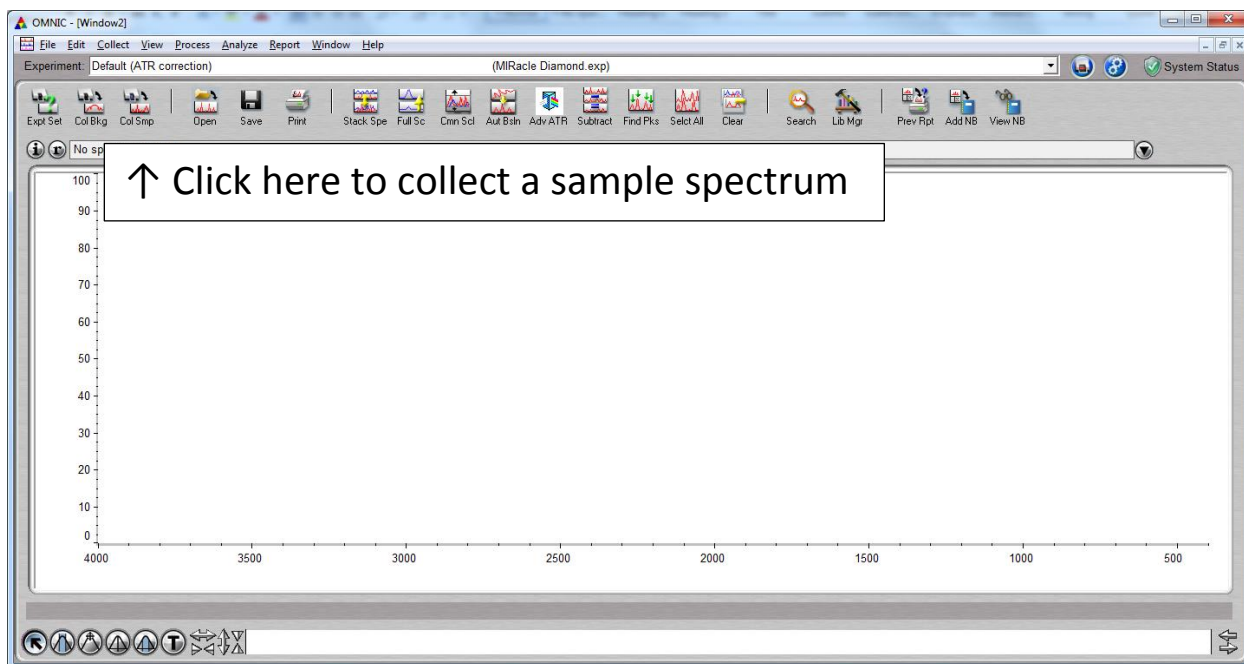


IV. Collecting a sample spectrum:

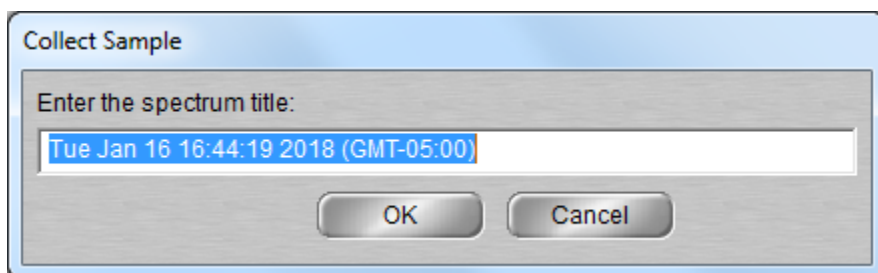
1. Before collecting a sample spectrum, you should first open a new display window. Click on the Window menu and select “New Window.” You will be prompted to name the window, but the default name provided is generally fine, otherwise select your preferred title. Click “OK” to accept the title. The Window menu is also used to move between open windows by clicking on the desired window name.



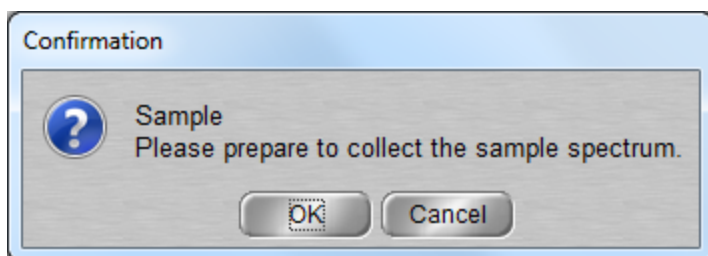
2. Click on the Collect Sample button in the upper left corner of the screen to collect a sample spectrum.



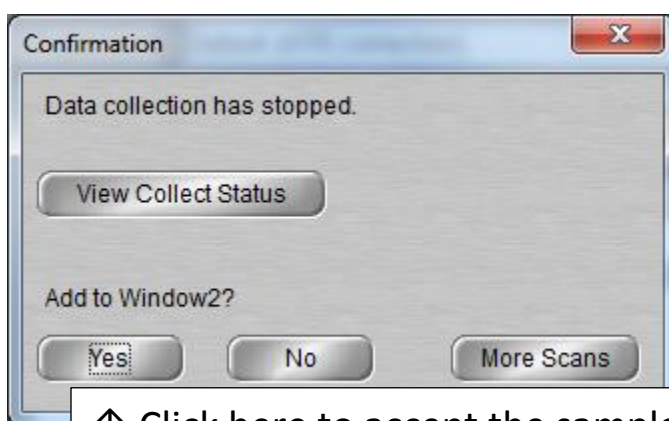
3. You will then be prompted to provide a title to display on the sample spectrum that is to be collected. Enter your desired title in the space provided, and click "OK." (Note: This is not the name of the file where this data will be saved. For how to name your data file, please see the section labeled "V. Saving your data.")



4. You will then be prompted to confirm that you want to collect a sample spectrum. (Note: if your sample is air sensitive or otherwise unstable, you can wait until this point to place your sample on the ATR crystal.) Click "OK" when you are ready.



5. After confirmation, the instrument begins to collect data. The steps that the instrument takes can be viewed in the bottom left corner of the screen. First, a couple of setup processes are completed, then a countdown of the chosen number of scans begins (1 of 8, 2 or 8, etc.). When the collection is complete, the spectrum is displayed and you are prompted to save the data to the currently opened window. If the data are acceptable, click "Yes" to save the spectrum.



↑ Click here to accept the sample spectrum and place it in the window

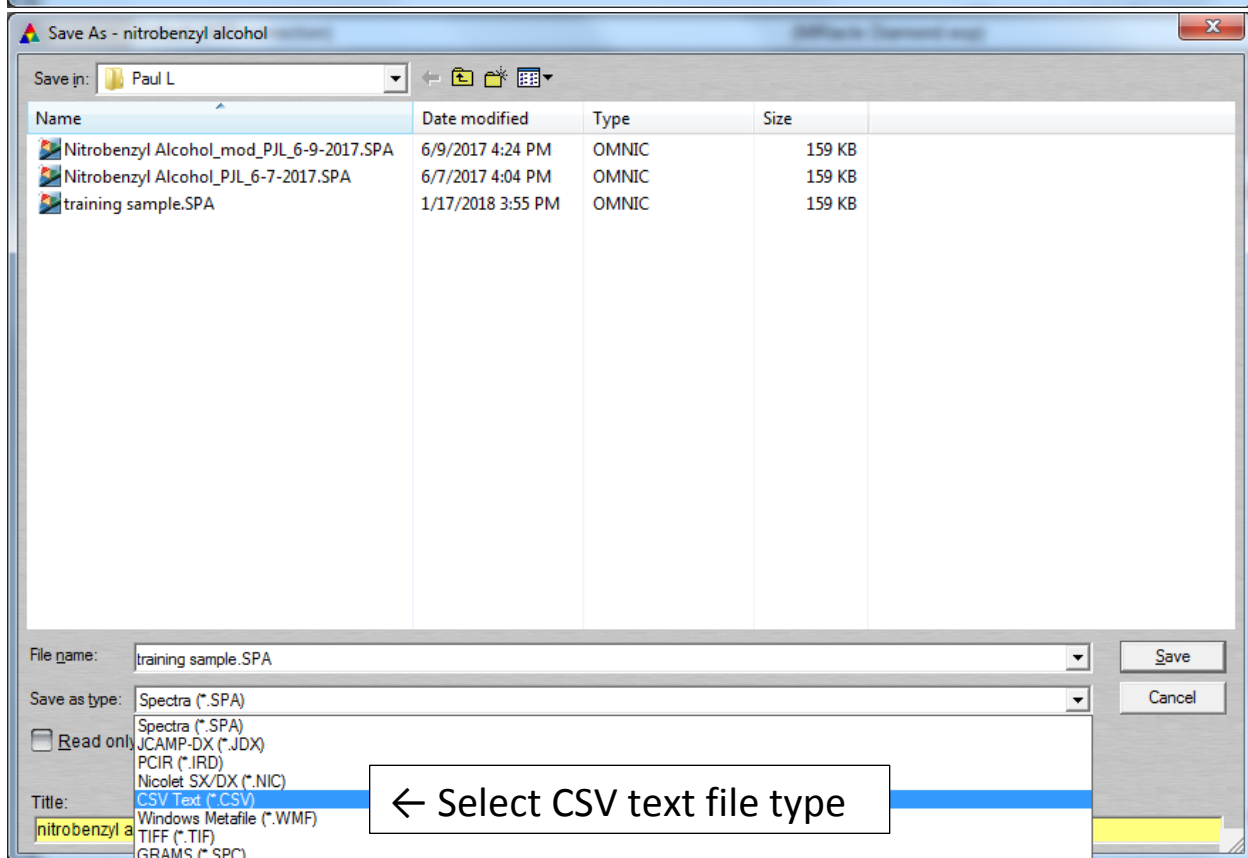
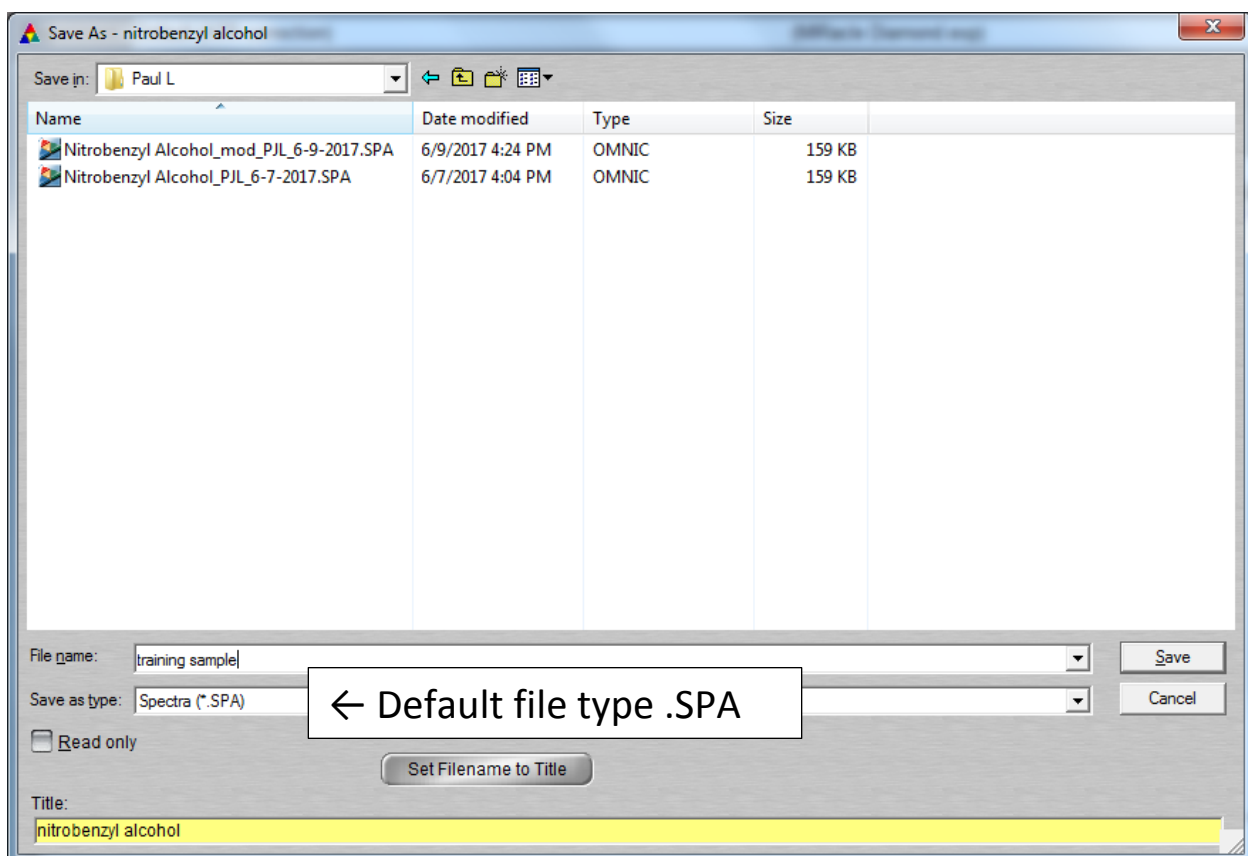
6. Mounting solid and liquid samples on the ATR crystal:

Both solid and liquid samples can be analyzed using the ATR accessory. It is important to have good contact between the sample and the ATR crystal for a good spectrum to be obtained. Liquids naturally make good contact, whereas, solids

may not, depending upon the size and shape of the particles. Therefore it is necessary to screw down the threaded assembly located above the ATR crystal (known as the anvil), to press the solid material against the crystal before taking its spectrum. (Note: The anvil does not need to be screwed down to obtain a background spectrum, even when solid samples will be analyzed.)

V. Saving your data:

- 1) Click on the File menu and click on Save As. When the dialog box comes up, you can navigate to the folder where you want to store the data.
- 2) Click on the Create New Folder icon to initially create a folder for your data or to create sub-folders within your main folder.
- 3) You will want to save your data in 2 formats: the default type called Spectra (.SPA), and the text type called CSV (comma separated values). The .SPA file can be opened by the Omnic software in order to display it at a later date, whereas, the .CSV type can be opened by Excel, on your own computer, in order to work up data without paying for your time. Both files should be saved to a USB drive or emailed to yourself.

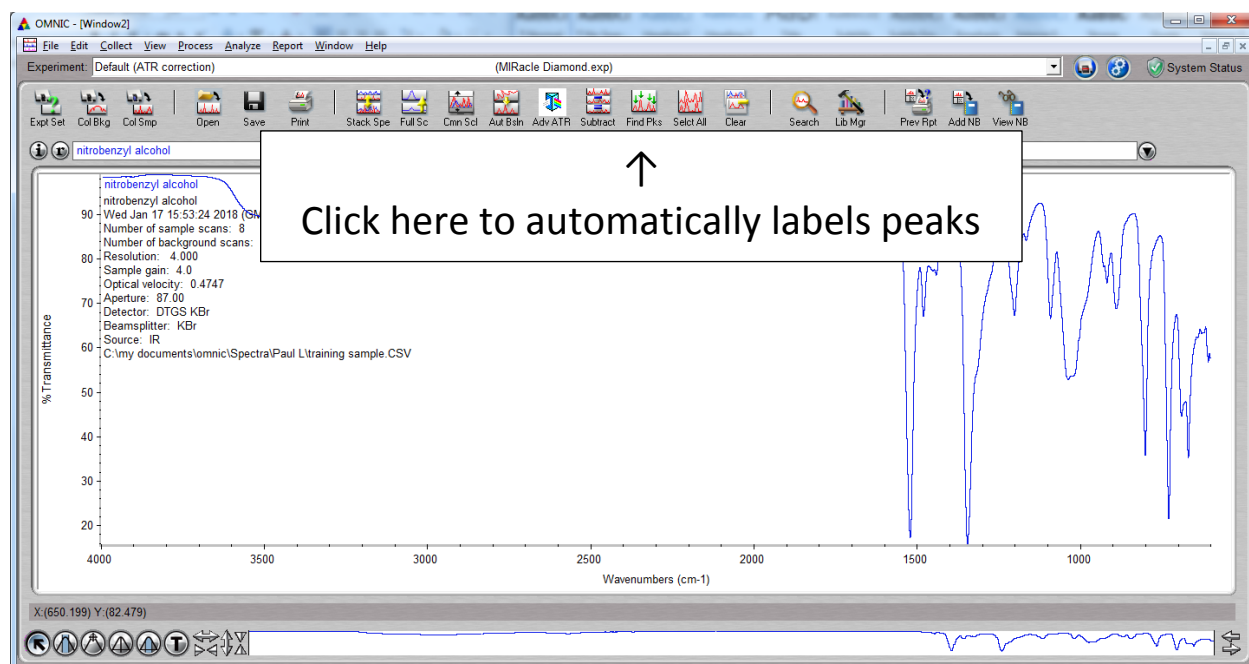


VI. Converting between % Transmittance and Absorbance

The default units of measurement for FTIR spectra are % Transmittance vs. Wavenumber. If you desire the spectrum intensity to be displayed in Absorbance units, click on the Process menu. The top 2 choices on this menu will toggle between % Transmittance and Absorbance. Some data work up processes (Subtraction, Baseline Correction and Library searches, among others), require that the data be expressed in Absorbance units.

VII. Peak Labeling

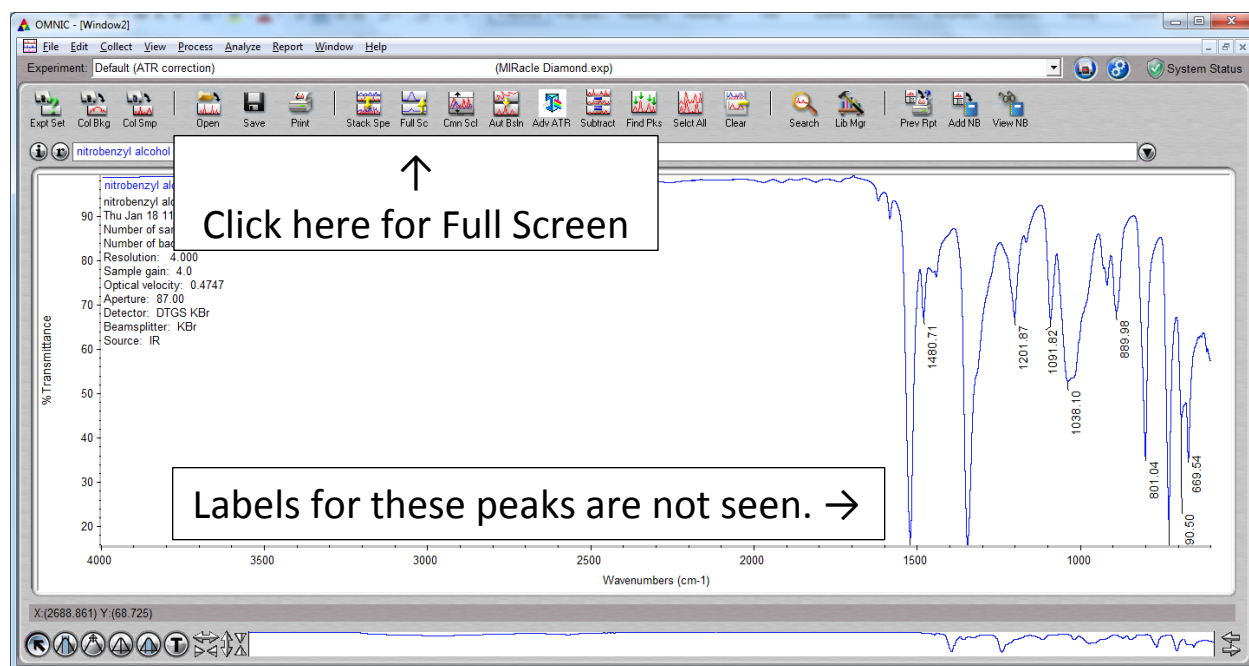
- 1) The wavenumber at which peaks are observed can be automatically labelled by clicking on the Find Peaks button on the Spectrum Toolbar.



- 2) When the Find Peaks button is clicked, a horizontal line appears on the spectrum, indicating the threshold value for defining a peak. Click with the tip of the cursor to reposition this threshold line. When the desired position is determined, click on the Replace button in the upper right corner of the page to lock in the threshold value.



- 3) The labels on some of the tallest peaks may not appear on the screen. This can be rectified by clicking on the Full Screen button on the toolbar.



- 4) Peak labels are not saved when spectra are saved. If you want a hardcopy of the labeled spectrum, you can print it out on printer next to the FTIR spectrometer. Alternately, you can copy and paste the labeled spectrum into a Word, Excel or PowerPoint document, or you can produce a report which will include the labeled spectrum, as well as, a listing of the peak labels. This report can be generated by going to the Report menu and clicking on Preview/Print Auto Report. This report can be copied and pasted to a Word, Excel or PowerPoint document and saved for future use.

VIII. Additional Topics:

The details of various types of Copying and Pasting, Subtracting Spectra, Baseline Correction and Library Searches will be covered individually during the training session.

IX. Shutting down the software:

When you are finished using the instrument, close the software and you will automatically be logged out of the Recharge software, and a dialog window will confirm that point. You are then no longer paying for time on the instrument.

X. Cleaning Sample Off of the ATR crystal:

The ATR assembly has some plastic parts that can be damaged by aggressive solvents like acetone or methylene chloride. We prefer that users use solvents like water, methanol or iso-propanol, if possible, to clean off their samples after analysis. If more aggressive solvents are required due to the nature of the sample, please try to restrict solvent contact to the crystal and stainless steel portions of the ATR assembly.