FT-IR Instrument Instructions
Paul Tschida and Terilyn Lawson

Introduction and Theory

Infrared (IR) spectrometry is based on molecular vibrations and rotations. These motions are accompanied by a change in the dipole moment of a molecule.[1] In theory, any molecule with a permanent or inducible dipole will be IR active. For instance, the homonuclear diatomics such as O_2, N_2, and H_2, have neither a permanent nor an inducible dipole moment and cannot be analyzed using IR spectrometry.[1] Other molecules, such as CO, clearly have a permanent dipole, with greater electron density around the oxygen and less electron density on the carbon.[1] Therefore, carbon monoxide is IR active. Finally, some molecules have inducible dipoles, which causes them to be IR active. For example, CO_2 has no permanent dipole, but a dipole can be induced if the molecule vibrates, with one oxygen atom moving towards the carbon and the other moving away from the carbon.

The IR induces these dipole changes by irradiating a sample, most often with a laser. If the frequency of radiation matches a natural frequency of vibration in the molecule, the energy is absorbed and a change in the amplitude of vibration occurs.[1] This change appears as a peak on the IR spectrum. As discussed above, any molecule with a dipole moment can be analyzed with the IR, regardless of the molecule's physical state (gas, liquid, or solid).[1]

Some of the more common stretching and bending motions in a molecule are shown in Figure 1.[1]

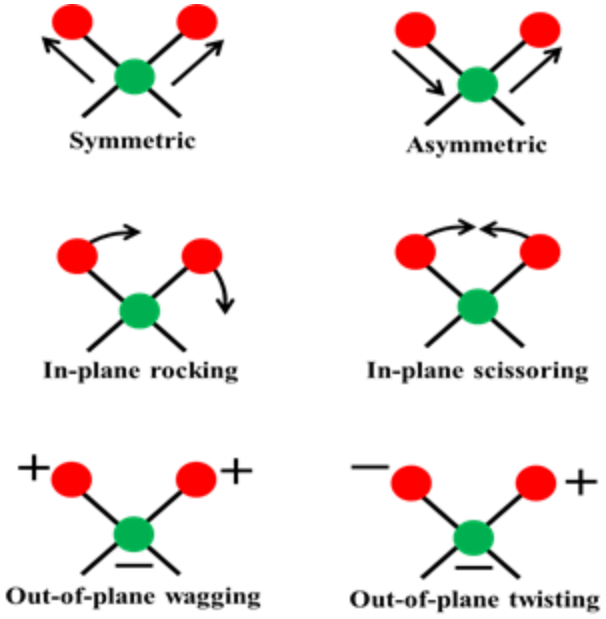


Figure 1. Types of Molecular Vibrations.
The + indicates motion out of the page and the – indicates motion into the page.

Block Diagram/Explanation

The IR available to Chem 413 students is a Nicolet 6700 Fourier Transform-Infrared Spectrometer (FT-IR). A FT-IR uses an interferometer to measure all IR frequencies simultaneously and produces an interferogram.[2] The interferogram is operated on mathematically by a Fourier Transformation, outputting an absorption or % transmittance spectrum. [2]

A detailed instrumental diagram of a single- beam FT-IR is shown in Figure 2.[1] An IR source in the near region (12800 to 4000 cm^{-1}), mid-region (4000-200 cm^{-1}) or far-region (200-10 cm^{-1}) sends IR radiation into the interferometer where it travels through the beamsplitter into a fixed or movable mirror.[1] Once the IR radiation impacts a mirror, the radiation travels into the sample compartment and to the IR transducer.[1] The two beams of radiation produced by the beamsplitter can interact with each other resulting in an interferogram.[2] Once the signal has impacted the transducer, the interferogram is Fourier Transformed into the resulting spectrum.[2] The laser is used as a calibration technique for the movable mirror in the interferometer.[3]

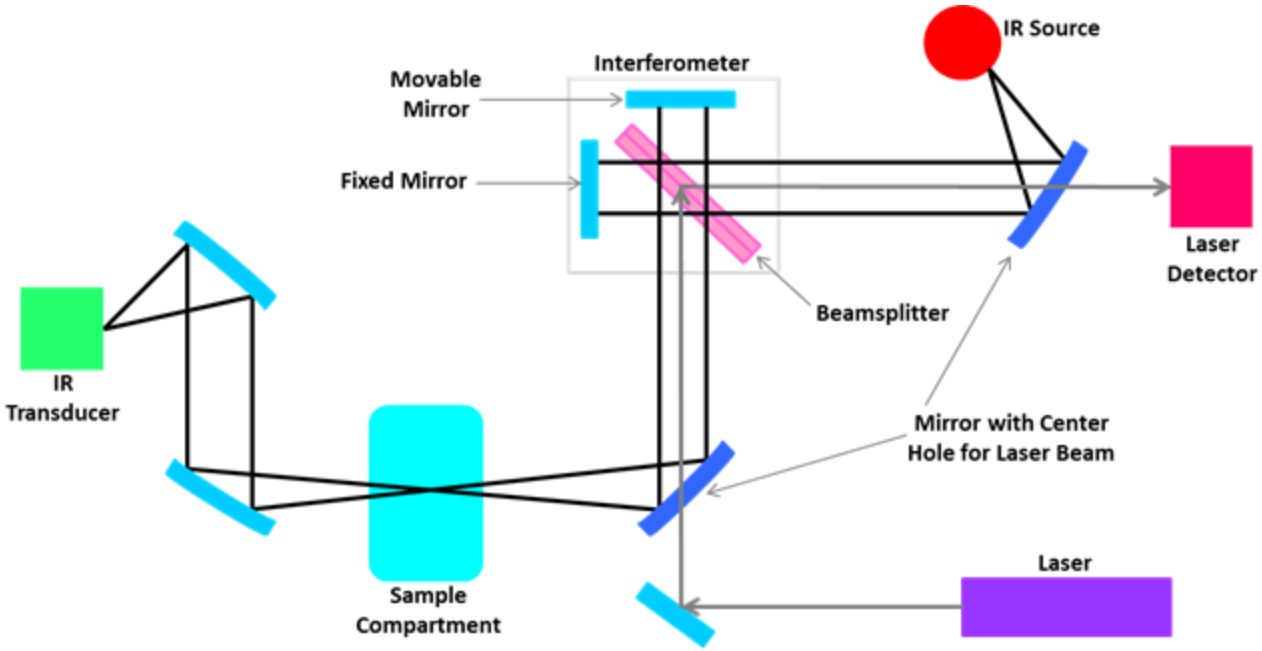


Figure 2. Instrumental diagram of a Single-Beam FT-IR spectrometer.[1]

The most common interferometer used in FT-IR spectroscopy is a Michelson interferometer (Figure 3).[1] When the IR radiation travels to the beamsplitter, it is either transmitted or reflected.[1] Half of the radiation beam will impact either a fixed or moving mirror and will be reflected back to the beamsplitter where beam interaction can occur.[1,2] The motion of the movable mirror causes the radiation to fluctuate when it reaches the detector.[1] Depending on the distance of the movable mirror, the fluctuation can be either destructive or constructive.[1] The difference in the path lengths of the two mirrors, M and F in

Figure 3, is called the retardation.[1] An interferogram is the retardation plotted against the output power of the detector. The interferogram will eventually get Fourier Transformed, outputting a spectrum.[1]

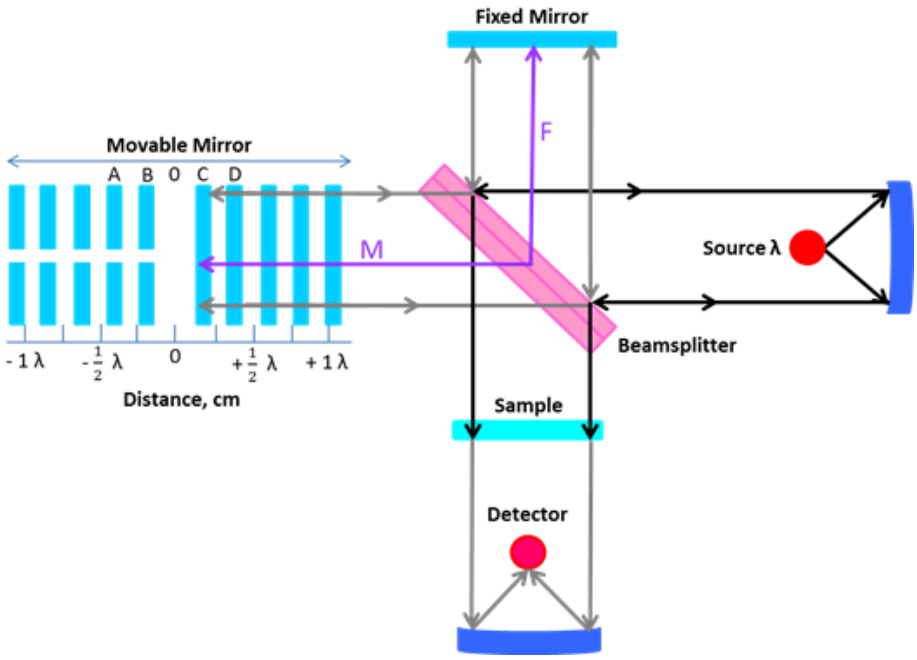


Figure 3. Illustration of a Michelson interferometer used in the FT-IR.[1]

An annotated instrument picture of the Nicolet 6700 FT-IR is also shown in Figure 4.[1] This instrument has a single beam IR and uses a Michelson interferometer. The IR source is Globar and provides mid-region IR radiation. The FT-IR laser is thought to be a He-Ne laser but further research is needed to confirm this. The beamsplitter is composed of KBr and the detector is a DTGS-KBr.

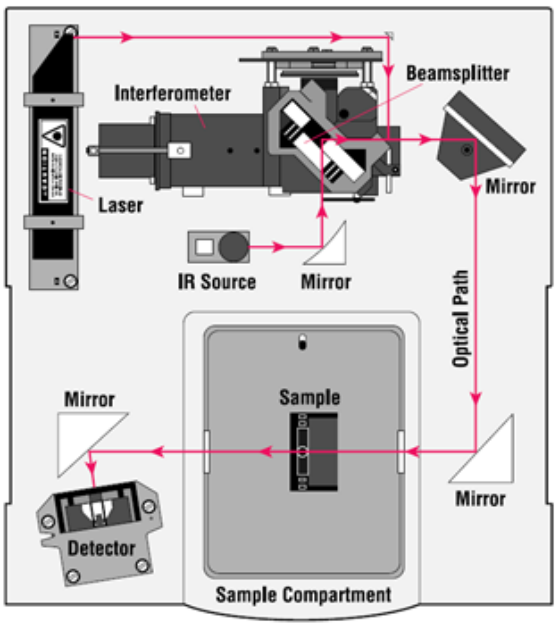


Figure 4. Annotated picture of the Nicolet 6700 FT-IR.[2]

Important instrumental parameters that should be recorded for the Nicolet 6700 FT-IR are the number of scans, resolution, data spacing, detector type, beamsplitter type, source type, accessory, range, gain, optical velocity, aperture and apodization function. The number of scans should be set at 128 or higher for better resolution. The resolution should be set at 2 and data spacing will vary depending on the number of scans and resolution. As stated in instrument picture, the FT-IR is composed of a DTGS KBr detector, a KBr Beamsplitter and an IR Globar source. The accessory, range, gain, optical velocity and aperture should be at Transmission ES, 400-4000 cm^{-1}, 1.0, 0.6329 and 25, respectively. The apodization function by default is Happ-General.

Instrument Operation

Safety Precautions

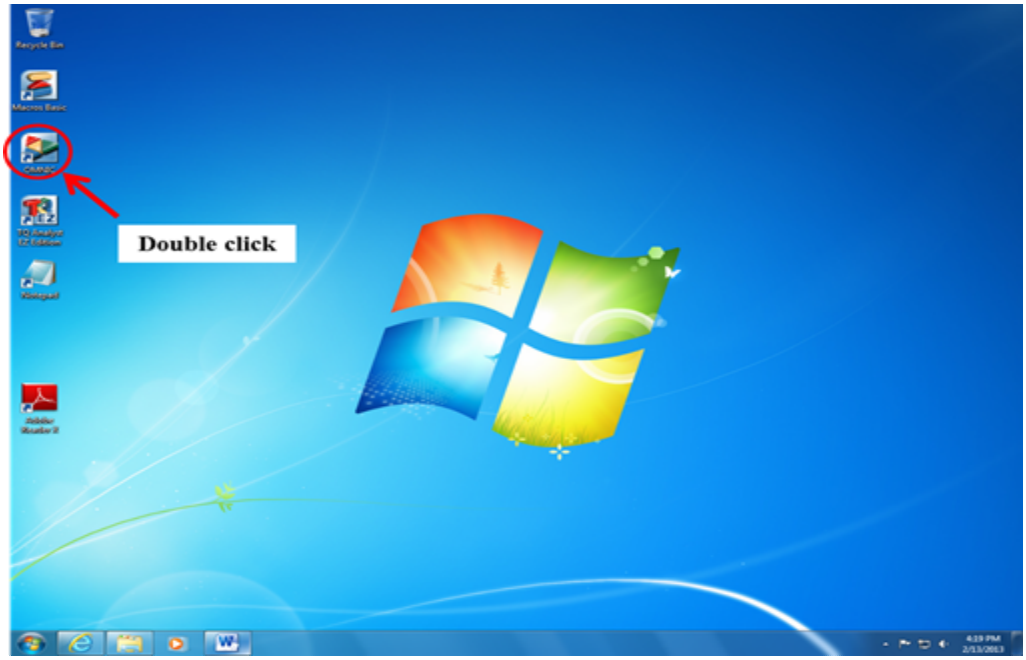
Some safety precautions to keep in mind when working with the FT-IR include: not using explosive or flammable samples, not placing anything on top of the electronics cover, not servicing the components yourself, ensuring that all cables are in good condition, and not staring at the laser or its bright reflection.[4] Normal operation of the instrument and common sense should prevent any unsafe situations.

Instrument startup

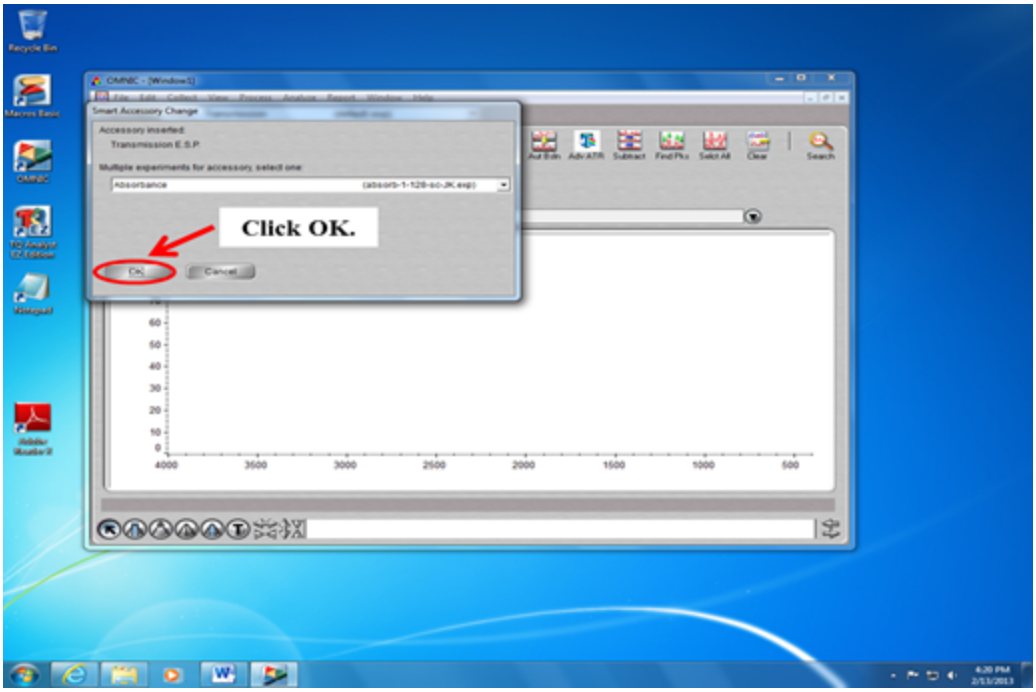
The instrument should have been left on by the previous user. To log in to the computer, choose the **ftiruser** profile and enter **ftiruser** as the password.

Instrument setup

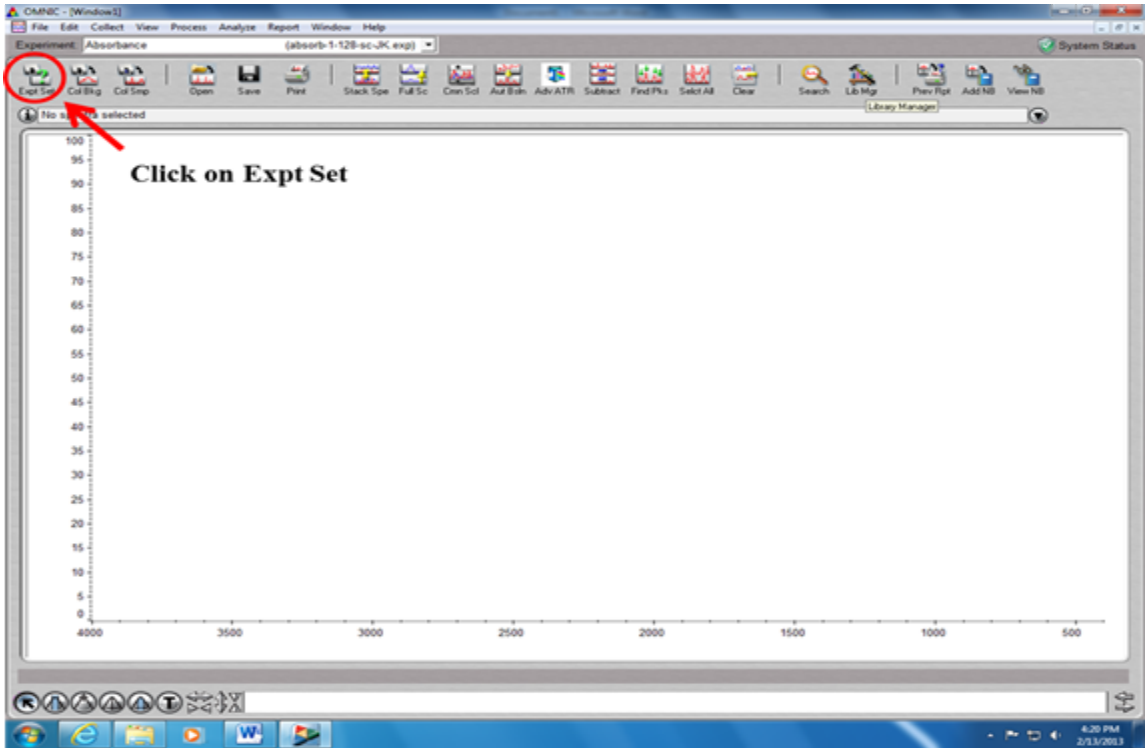
Double click on the OMNIC icon to open the FT-IR software.



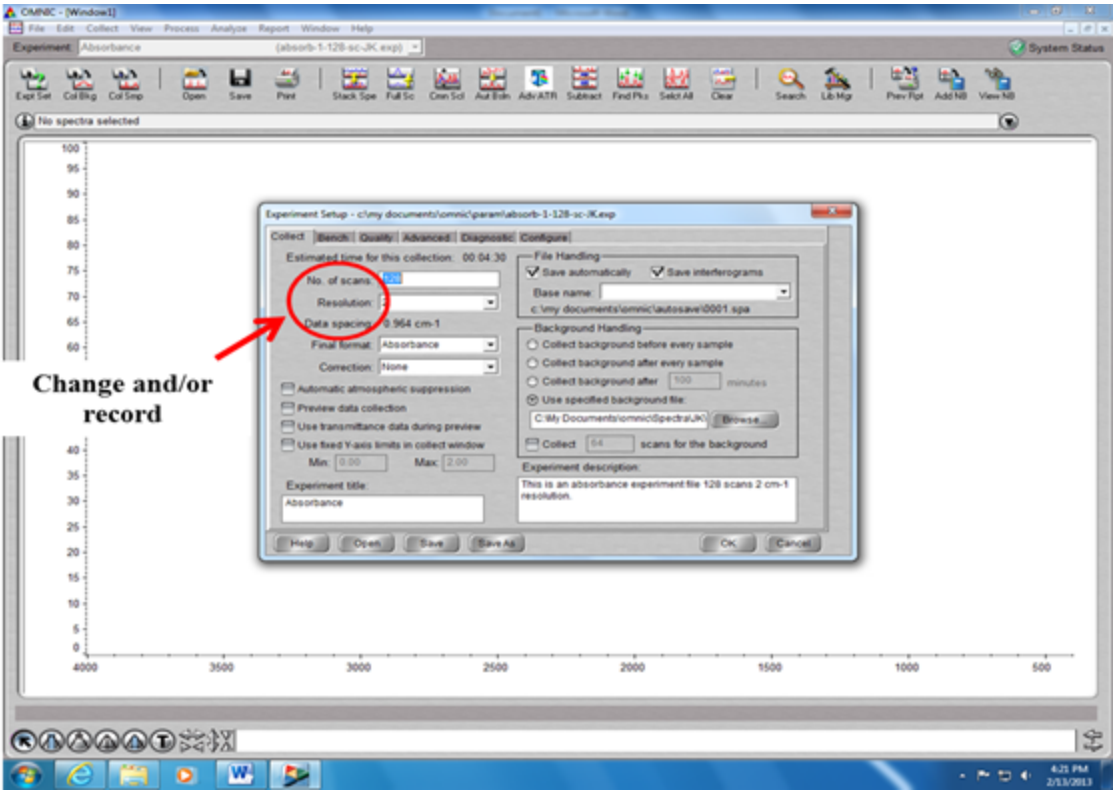
Click OK when the Smart Accessory Change box appears.



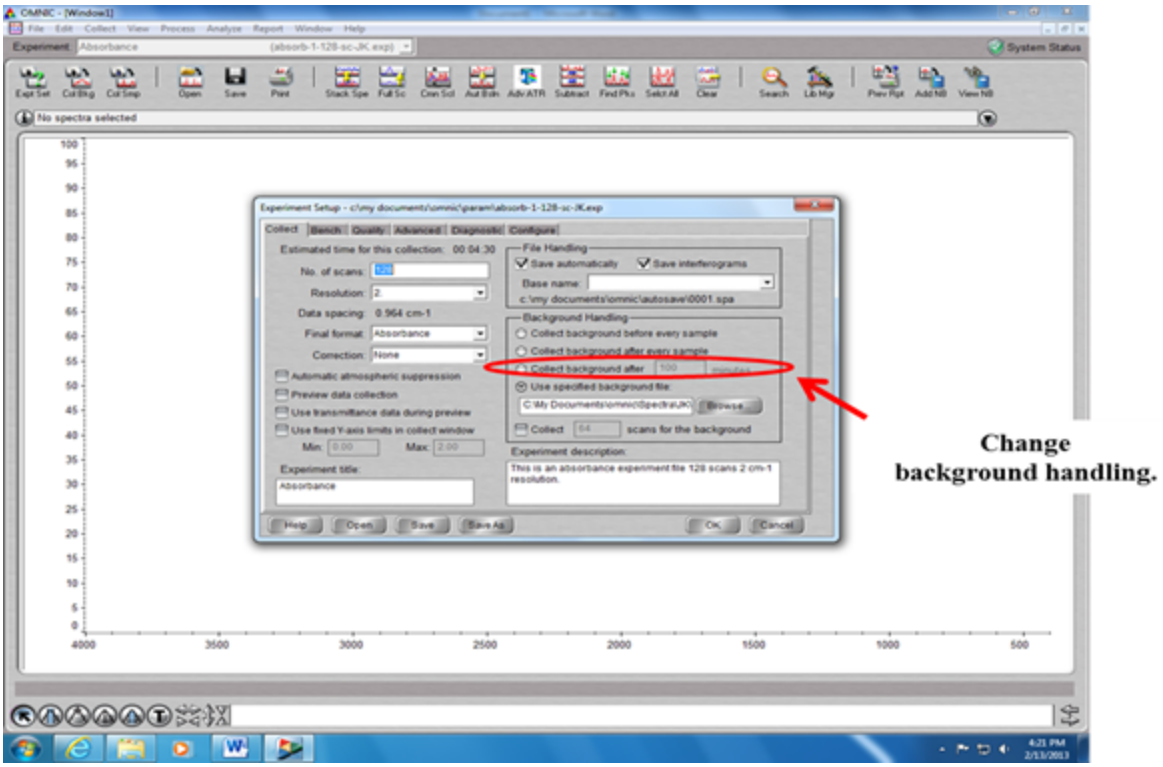
Click on Expt Set to prepare the instrument parameters.



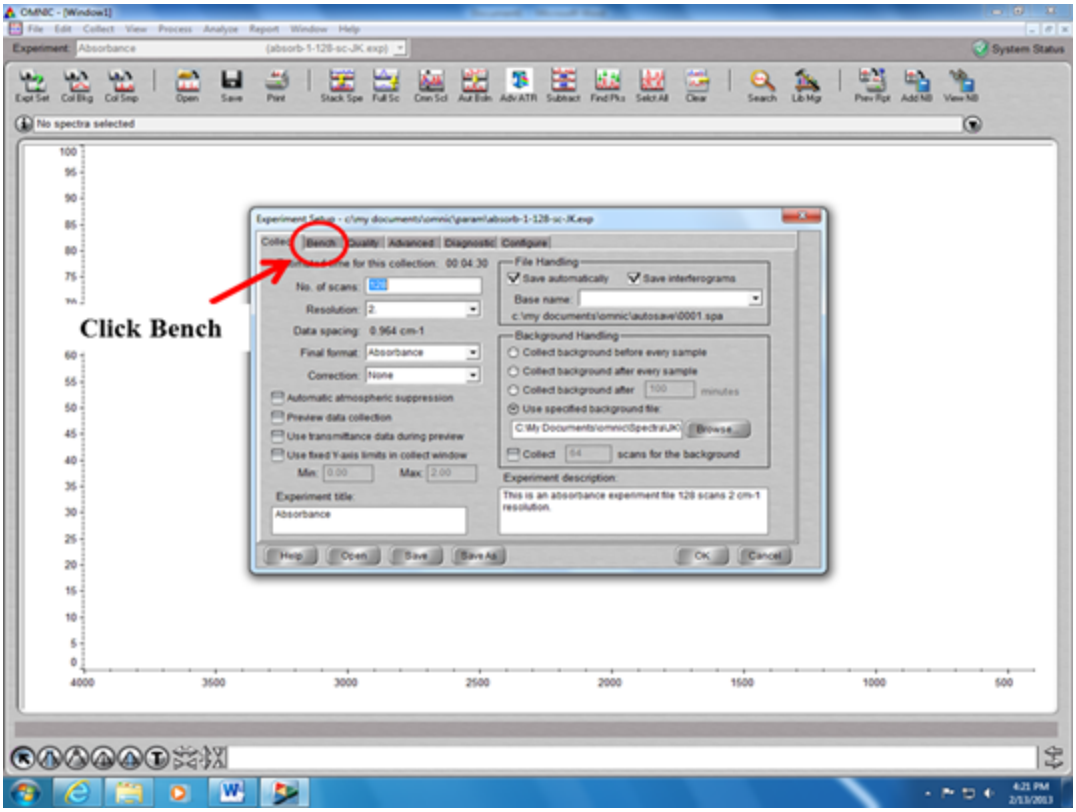
Change and/or record the number of scans, resolution, and data spacing.



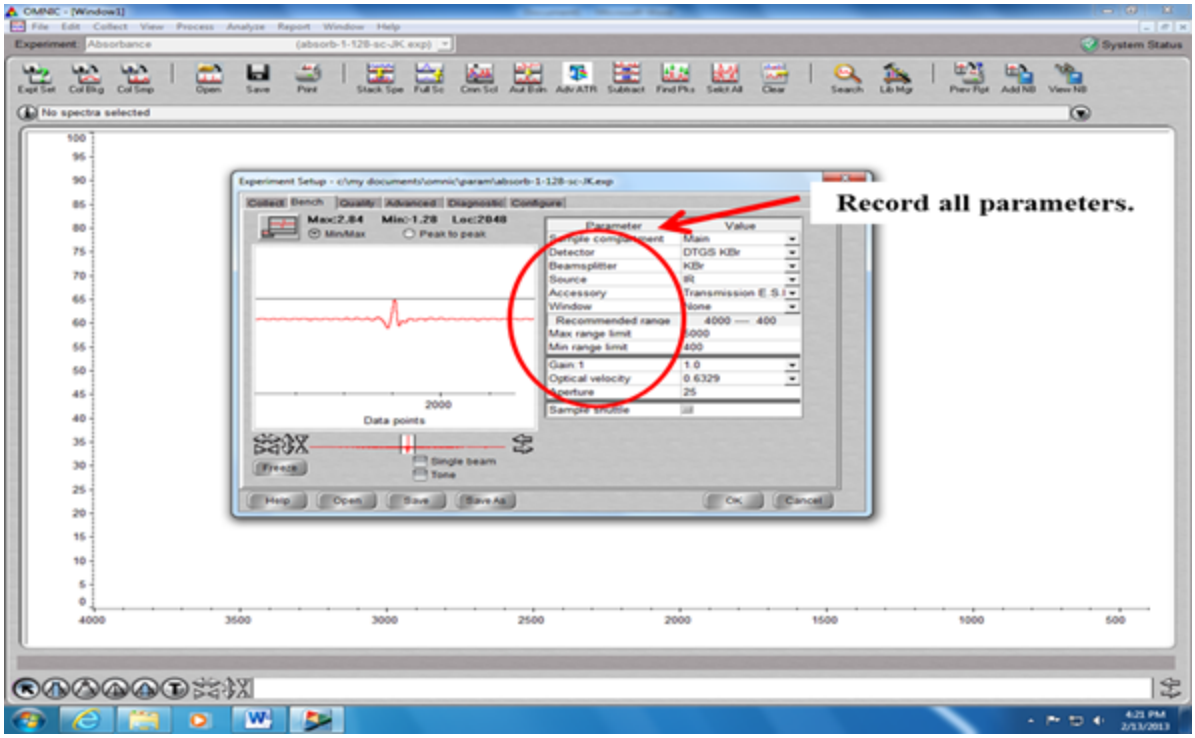
Change background handling to collect background after 100 minutes.



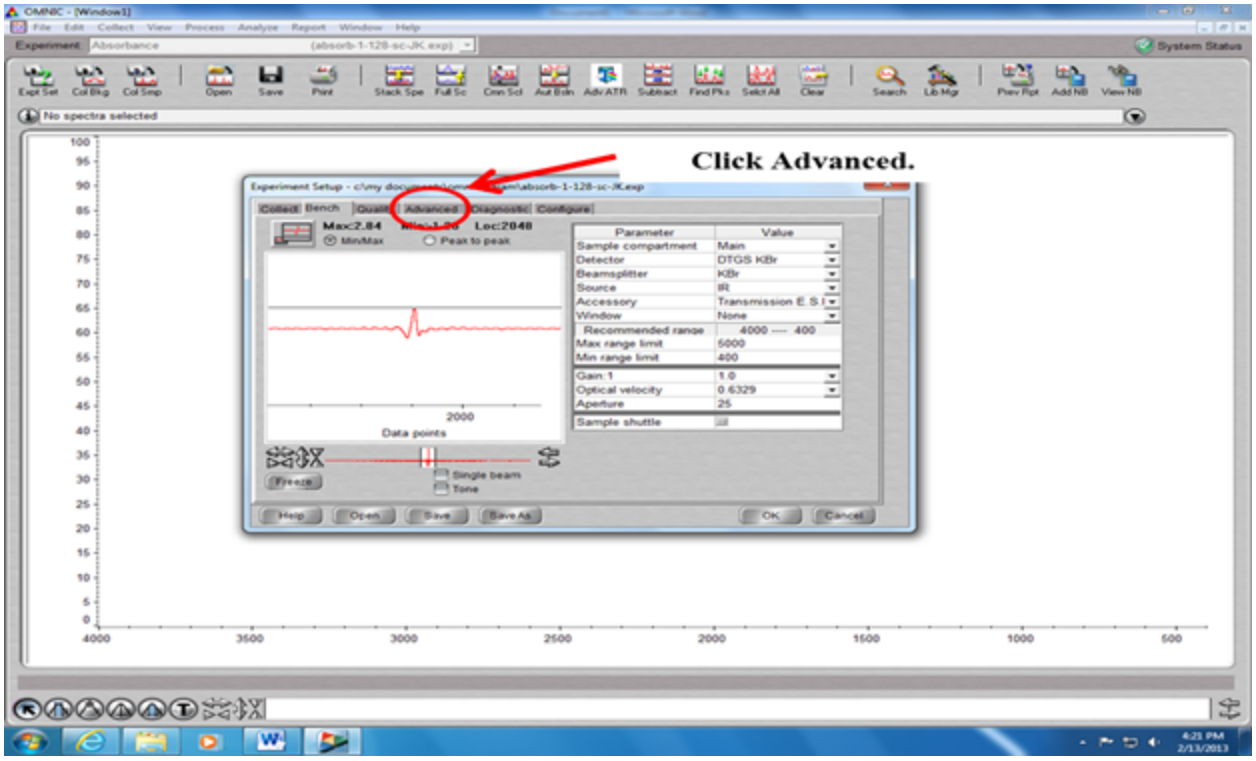
Click on the Bench tab.



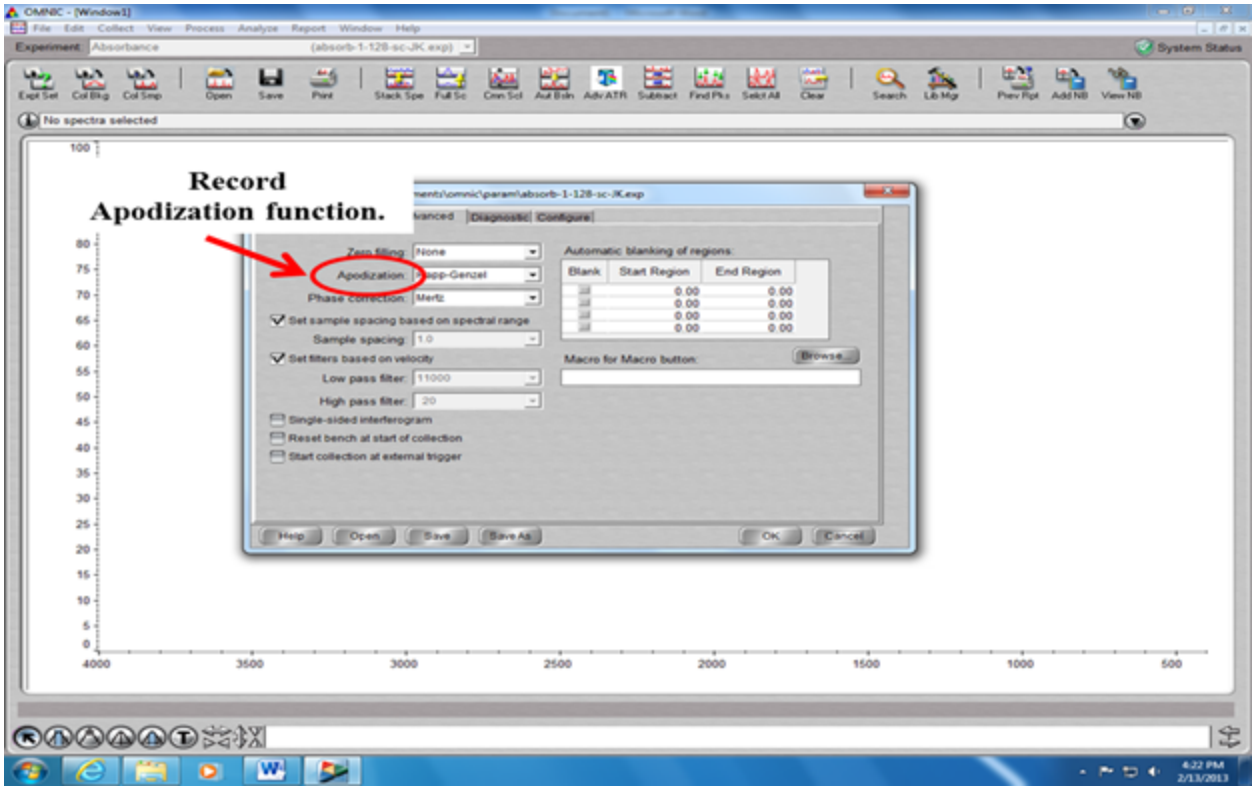
Record: detector, beamsplitter, source, accessory, range, optical density, and aperature.



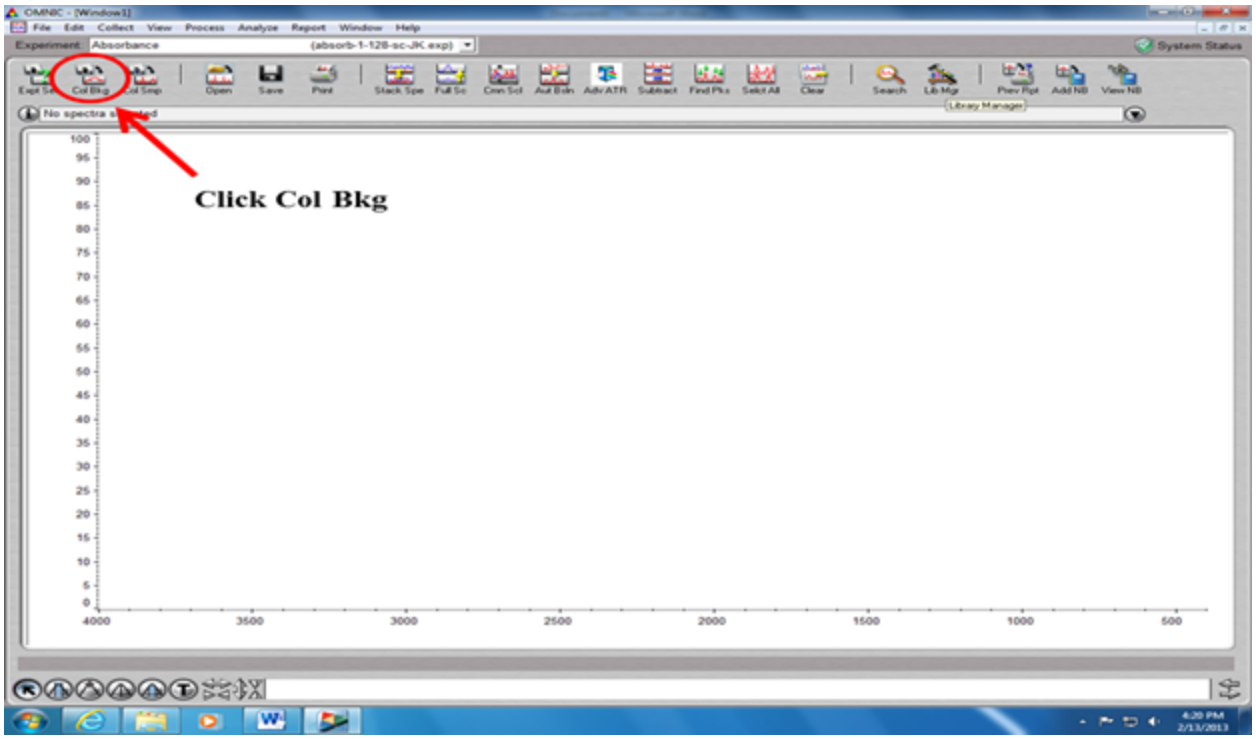
Click on the Advanced tab.



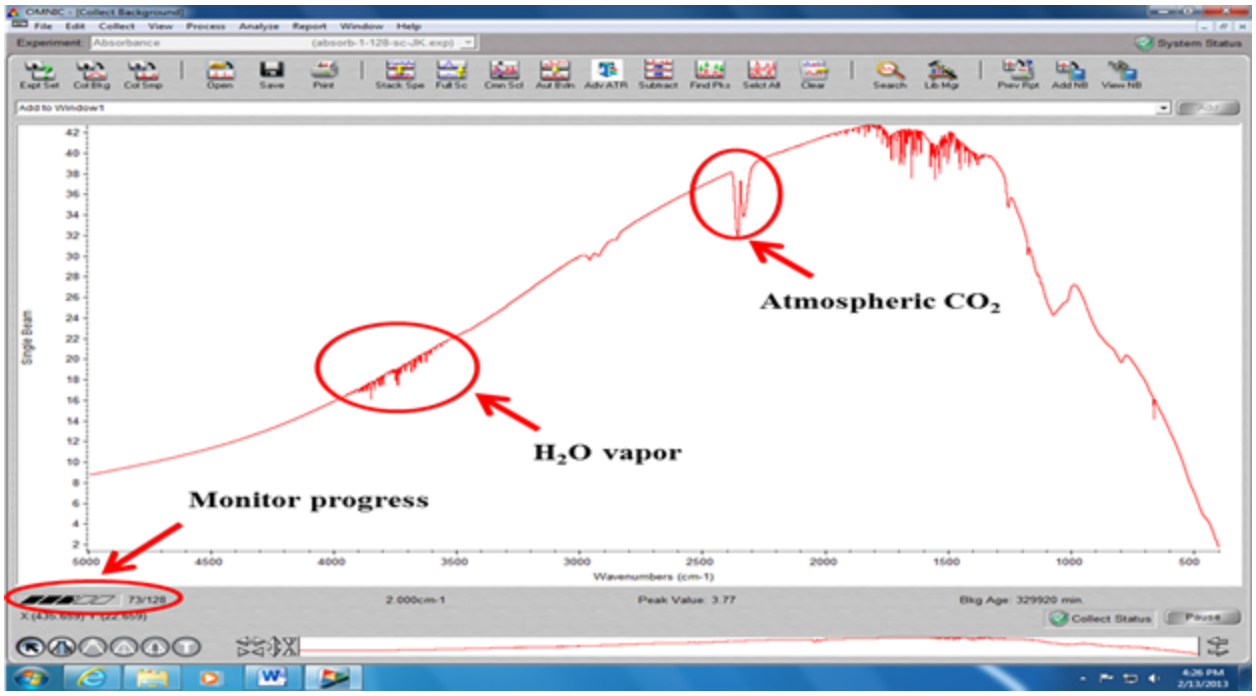
Record the Apodization function. Then click OK.



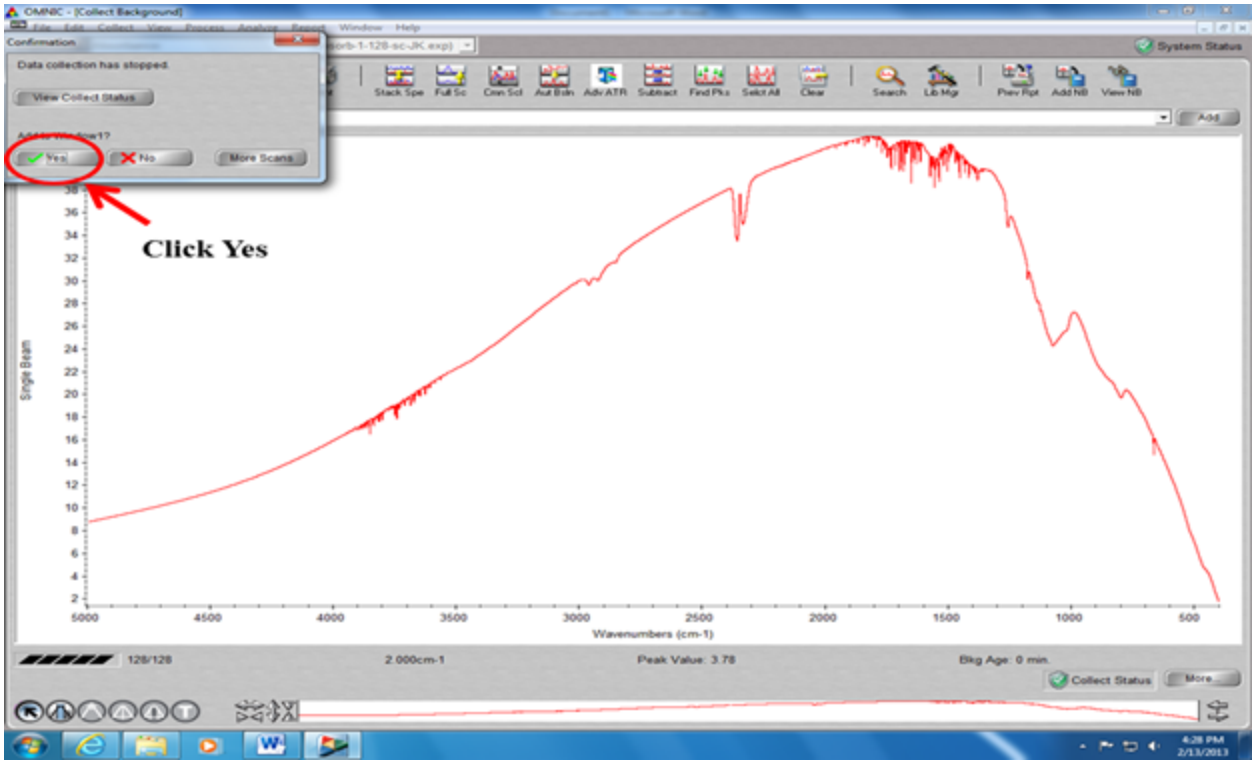
Make sure the sample compartment is closed and locked. Click on Col Bkg to collect the background spectrum. Click OK when the Confirmation box appears.



This is a typical background spectrum. Note the H_2O and CO_2 peaks. You can monitor the collection progress in the lower left.



Once the background spectrum has finished collecting, click Yes in the Confirmation box.



Sample Preparation

For liquids, place a small drop of the sample between two NaCl plates.[1] Put the plates into a sample holder and place into the path of the laser beam.[1]

For gases, let the sample expand in an evacuated cylindrical cell with windows suitable for IR analysis.[1]

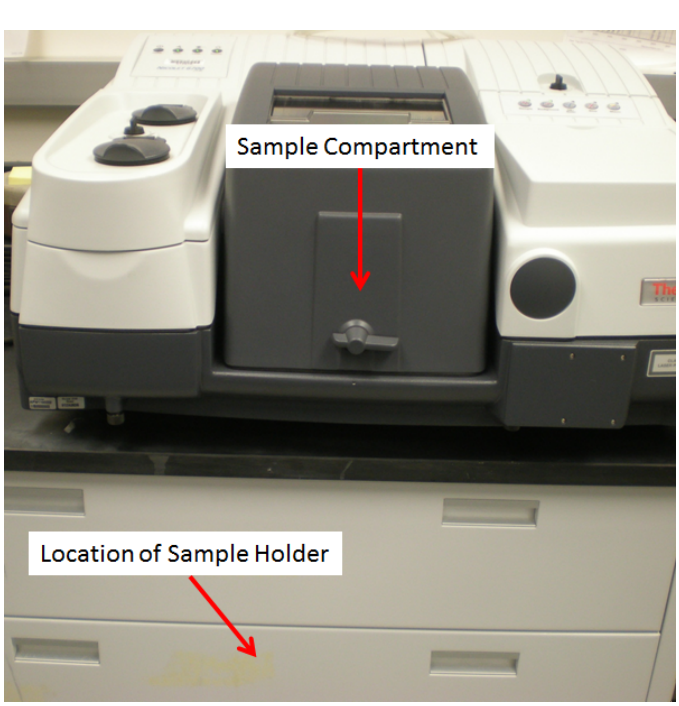
For solids, a variety of methods can be used. The most common is the use of a KBr pellet. A milligram or less of sample is mixed with 100 g of dried KBr powder in a mortar and pestle.[1] The mixture is then compressed with screws in a small anvil to yield a transparent disc.[1] The anvil can then be placed in a sample holder and the spectrum can be collected.

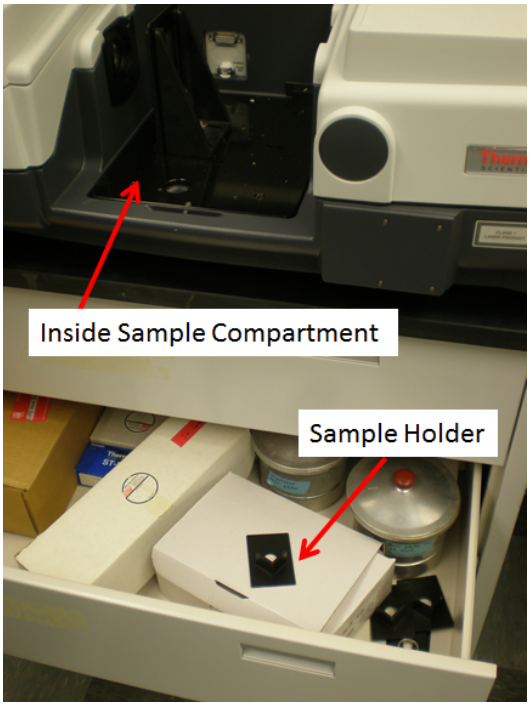
Another method for preparing solid samples involves dispersal of the sample in a mineral oil or a fluorinated hydrocarbon mull.[1] A mull is made by mixing ~5 mg of powdered sample with a couple of drops of Nujol, a heavy hydrocarbon oil.[1] The mull is then spread between two NaCl plates and the sample can be analyzed.[1]

For the plastic analysis, please visit the class website http://instrumentalanalysis.community.uaf.edu/techniques/instruments/ to watch a sample preparation

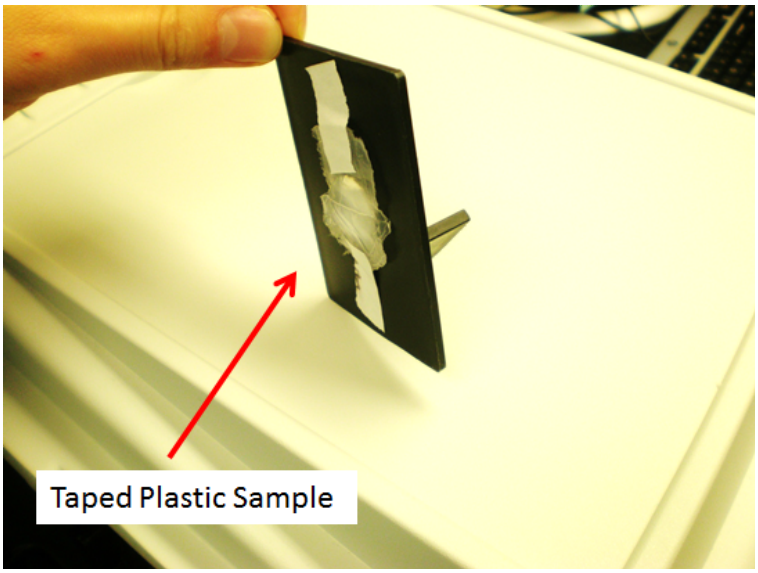
video. To access the video, hover over the techniques tab and click on the instrument information tab. The video is located under the Nicolet FT-IR header which is in bold font.

To place the sample in the instrument, open the sample compartment and find a sample holder. The sample holders are located in the second drawer under the instrument.

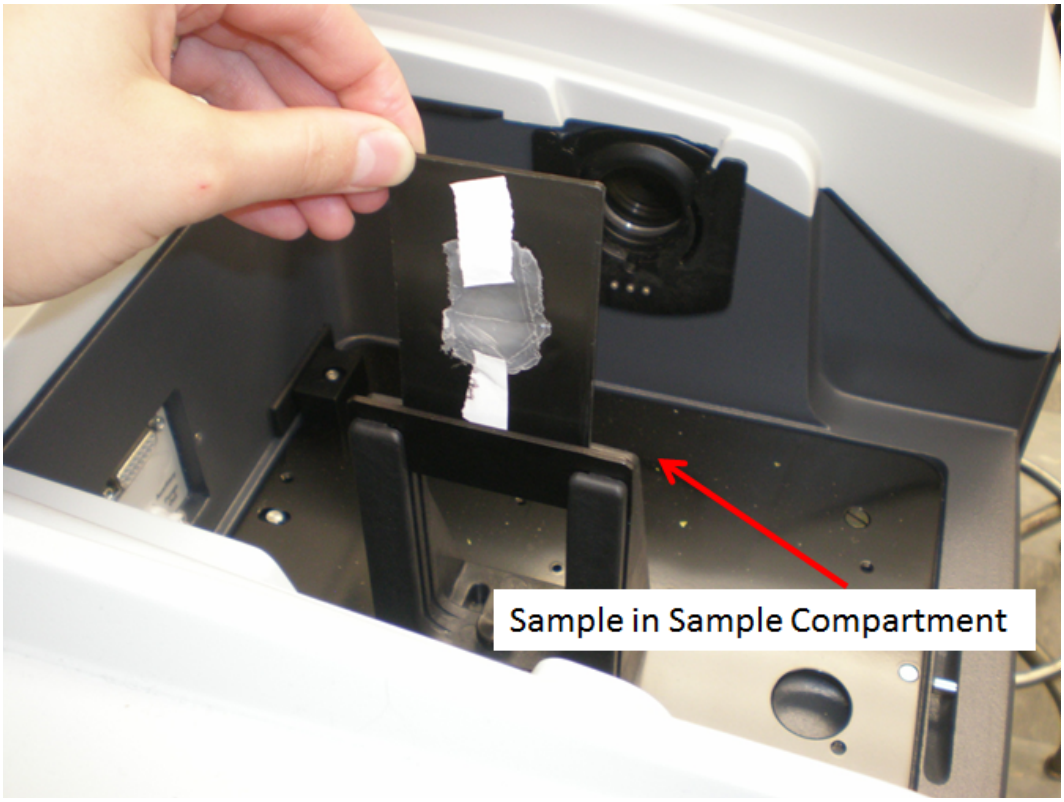




Tape your plastic sample to the back of the sample holder, making sure the sample is covering the window and/or the sample is aligned with the IR source.

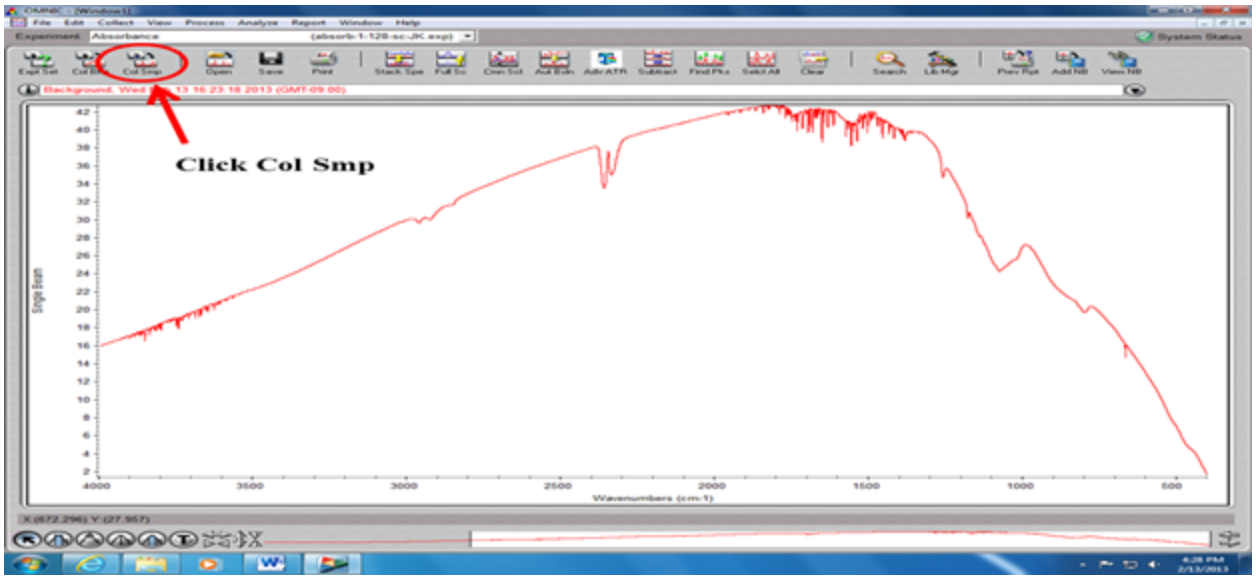


Insert the sample in the instrument, make sure the sample is covering the window, and ensure that the sample is aligned with the IR source.

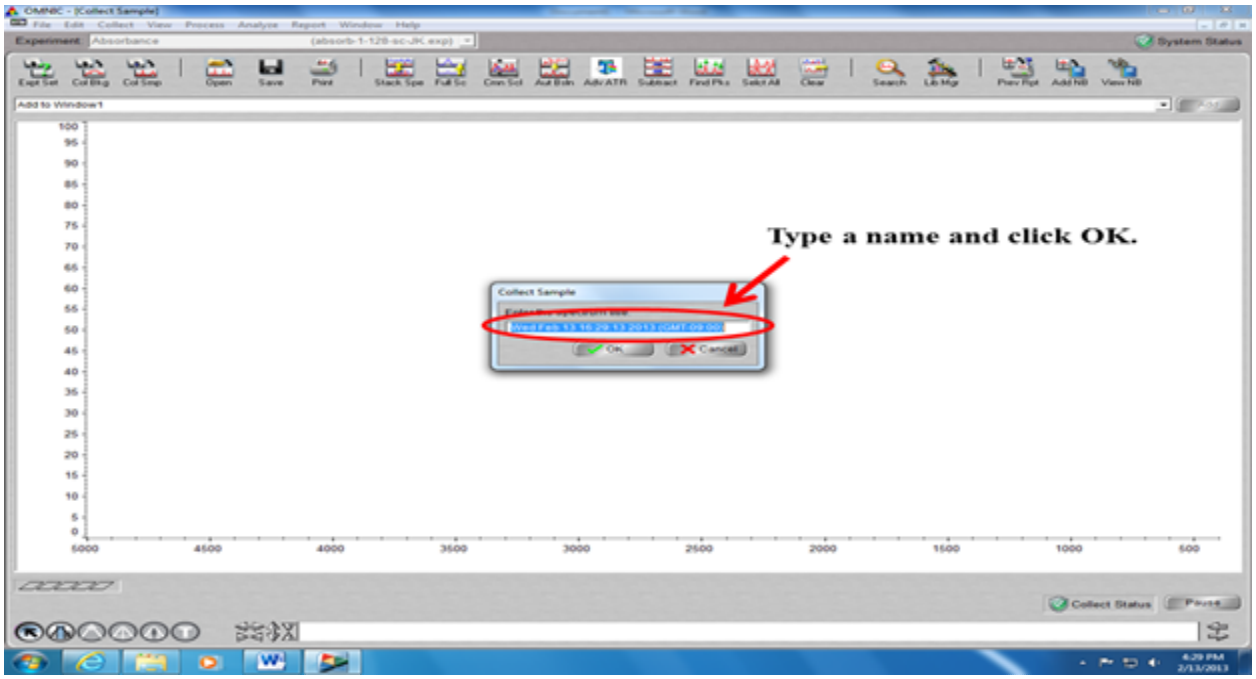


Instrument Operation

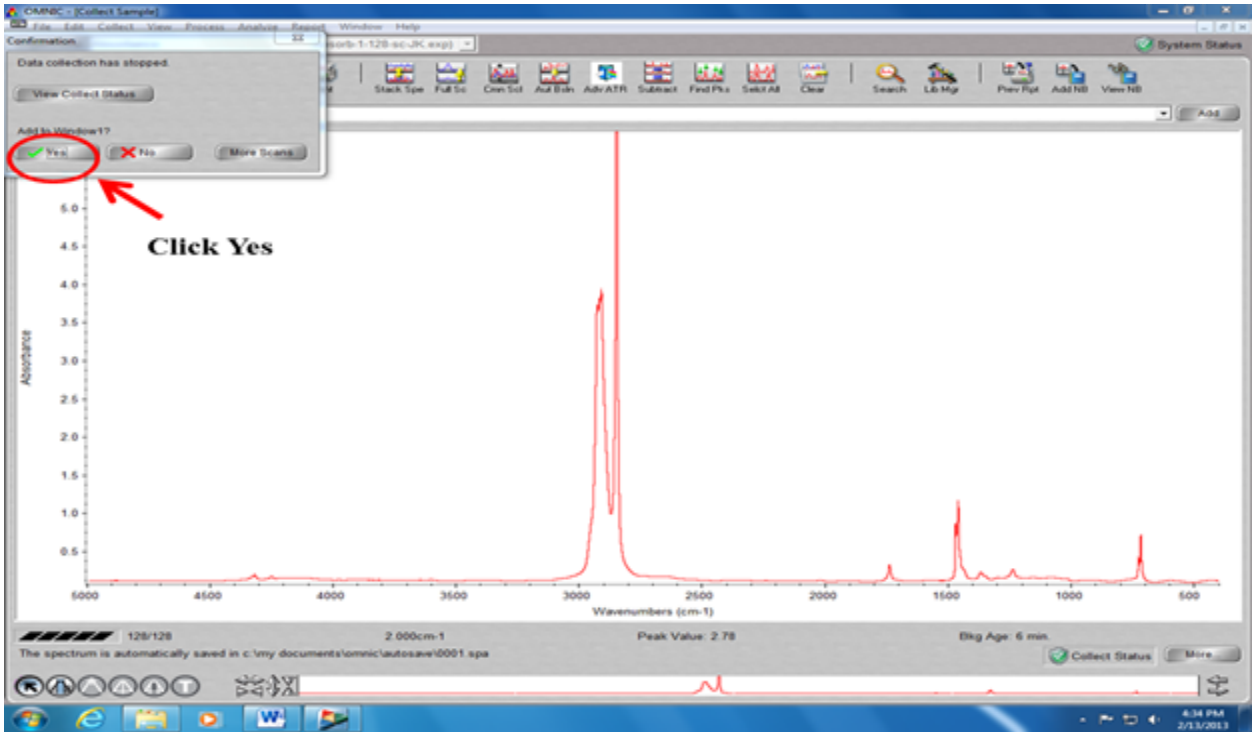
With the sample in place and the sample compartment closed and locked, click Col Smp.



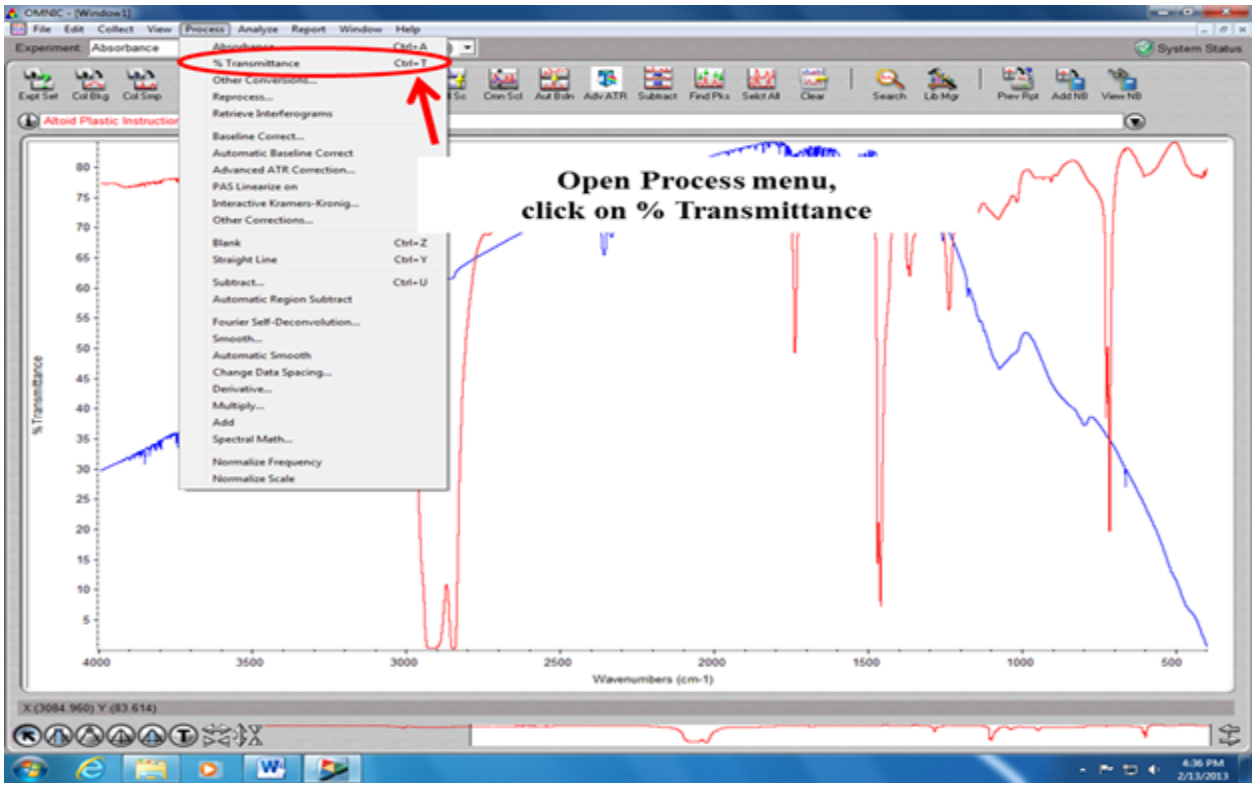
Name your sample spectrum and be sure to record it in your notebook. Then click OK. Click OK when the Confirmation box appears.



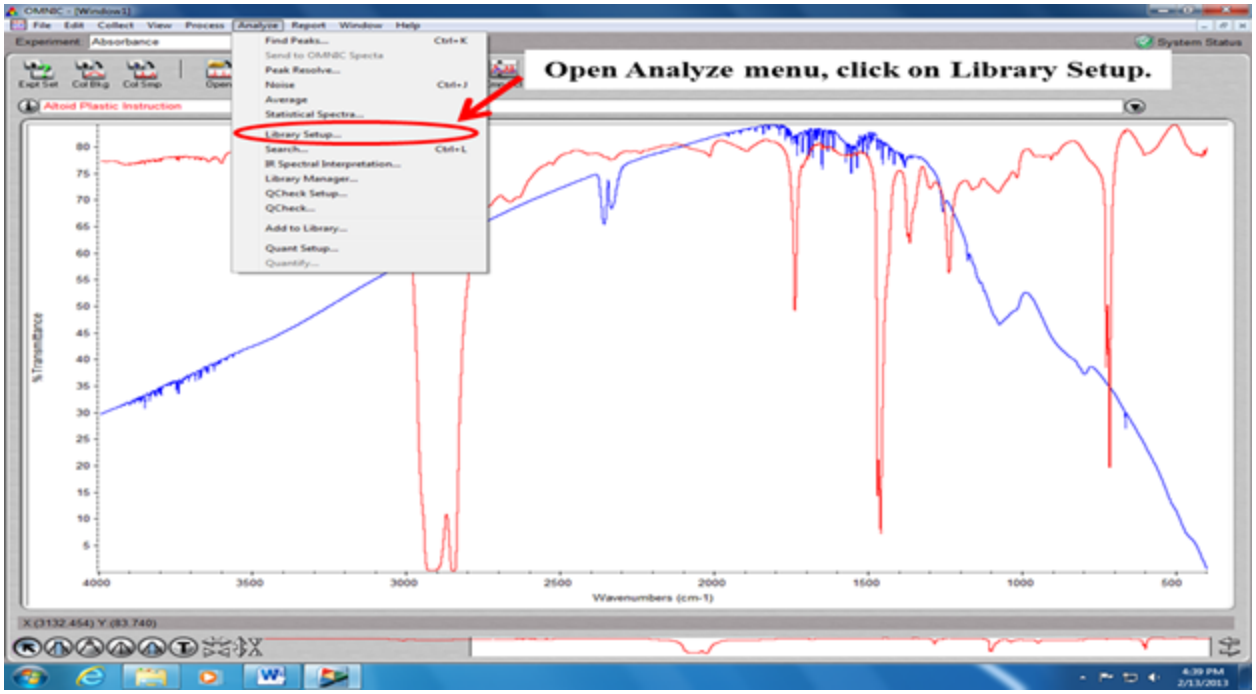
When your sample is done collecting, click Yes to add it to the window.



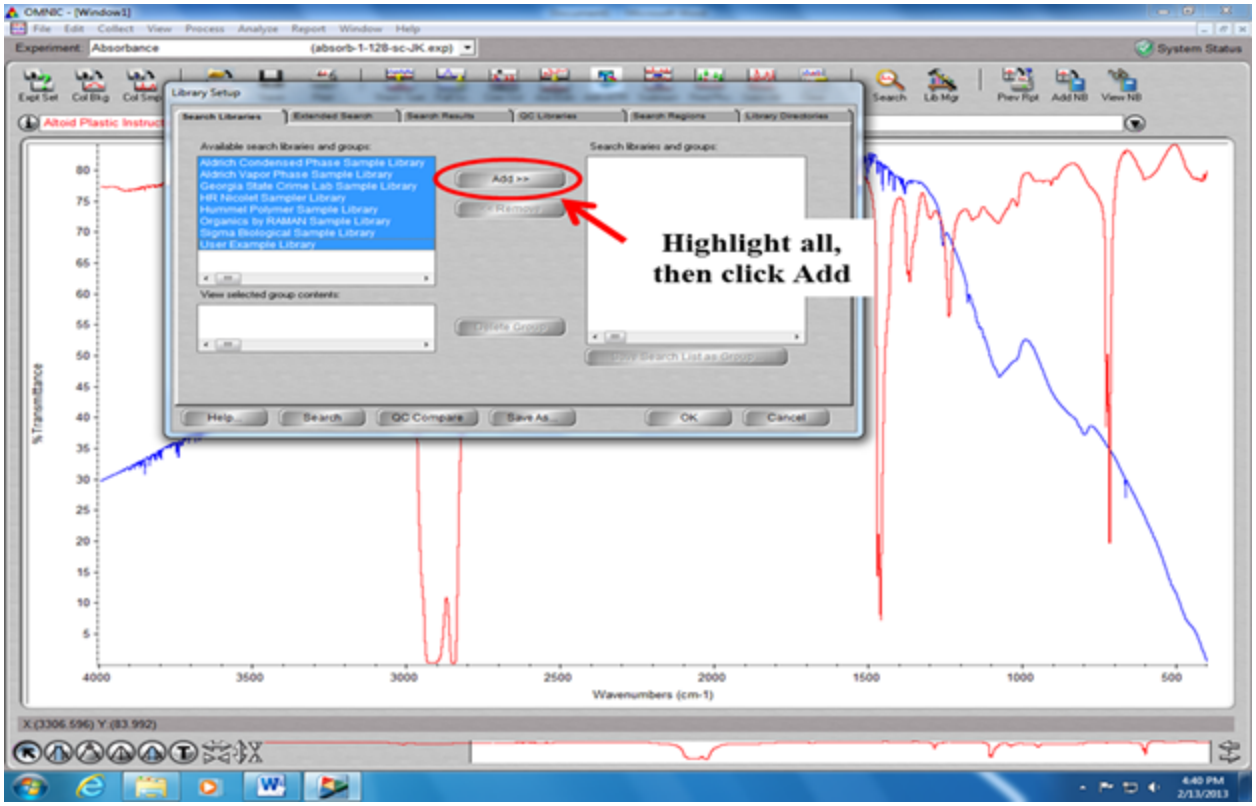
To change the y-axis from Absorbance to % Transmittance, open the Process menu and click on % Transmittance.



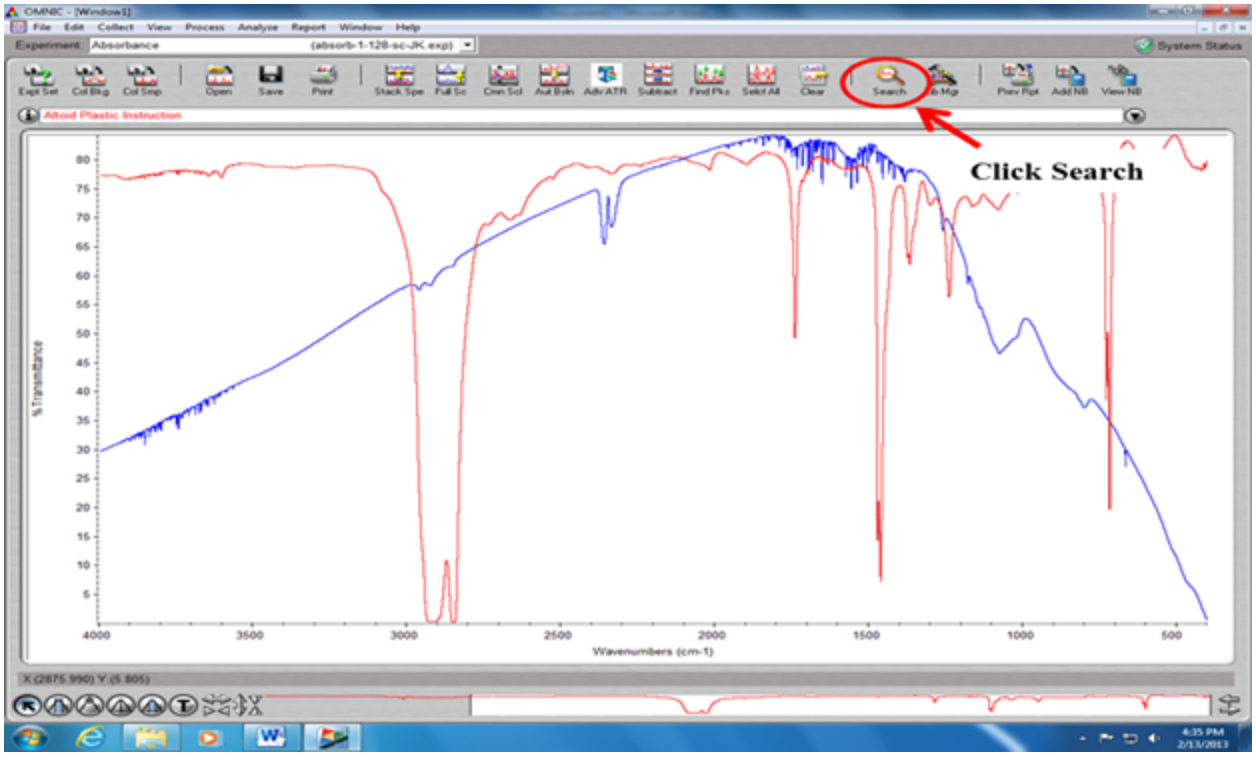
To match your sample to the instrument's spectral libraries, open the Analyze menu and click on Library Setup.



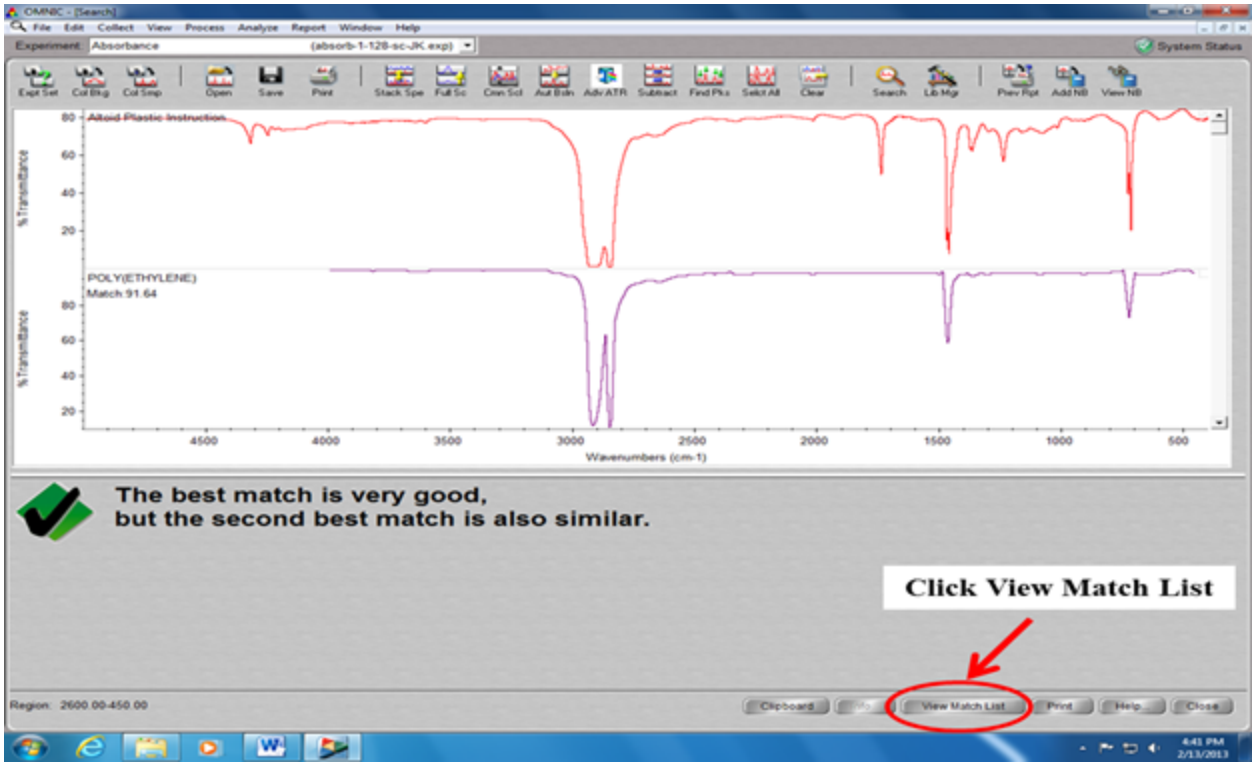
Highlight all of the libraries and click Add. Then click OK.



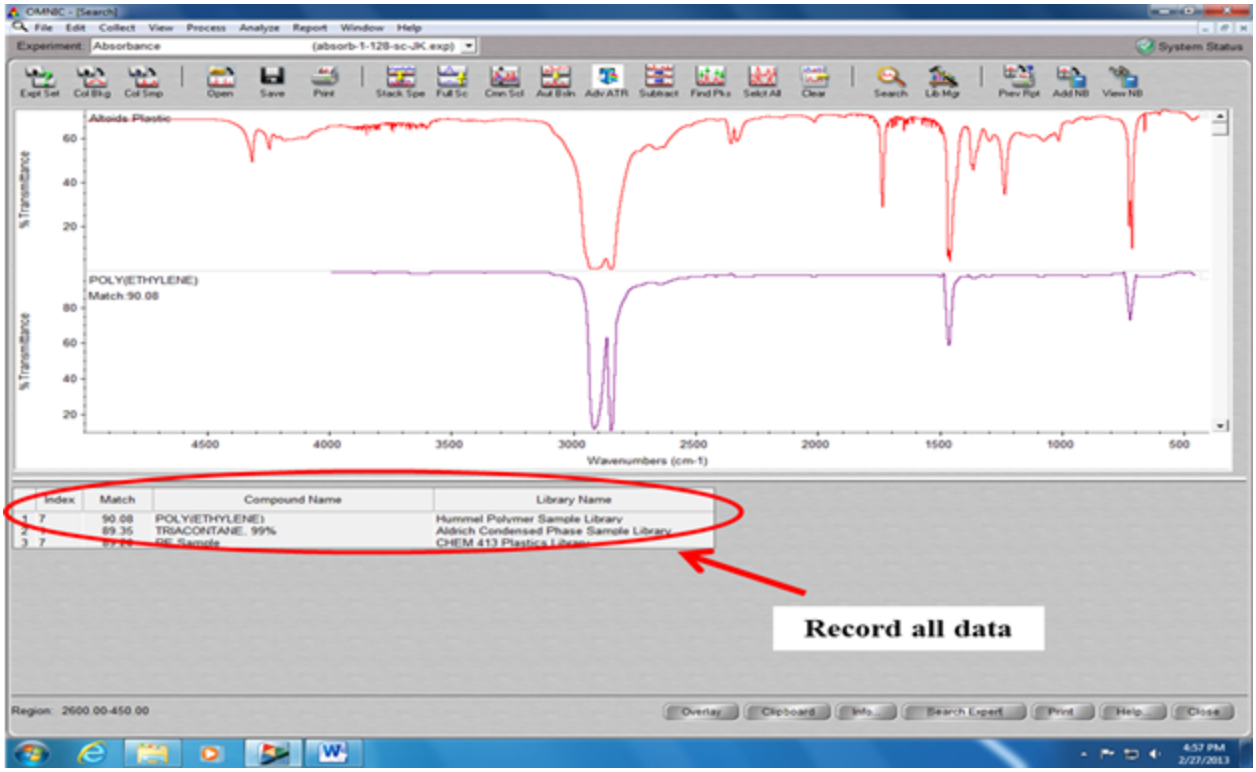
Click on Search to match your sample spectrum to the spectral libraries.



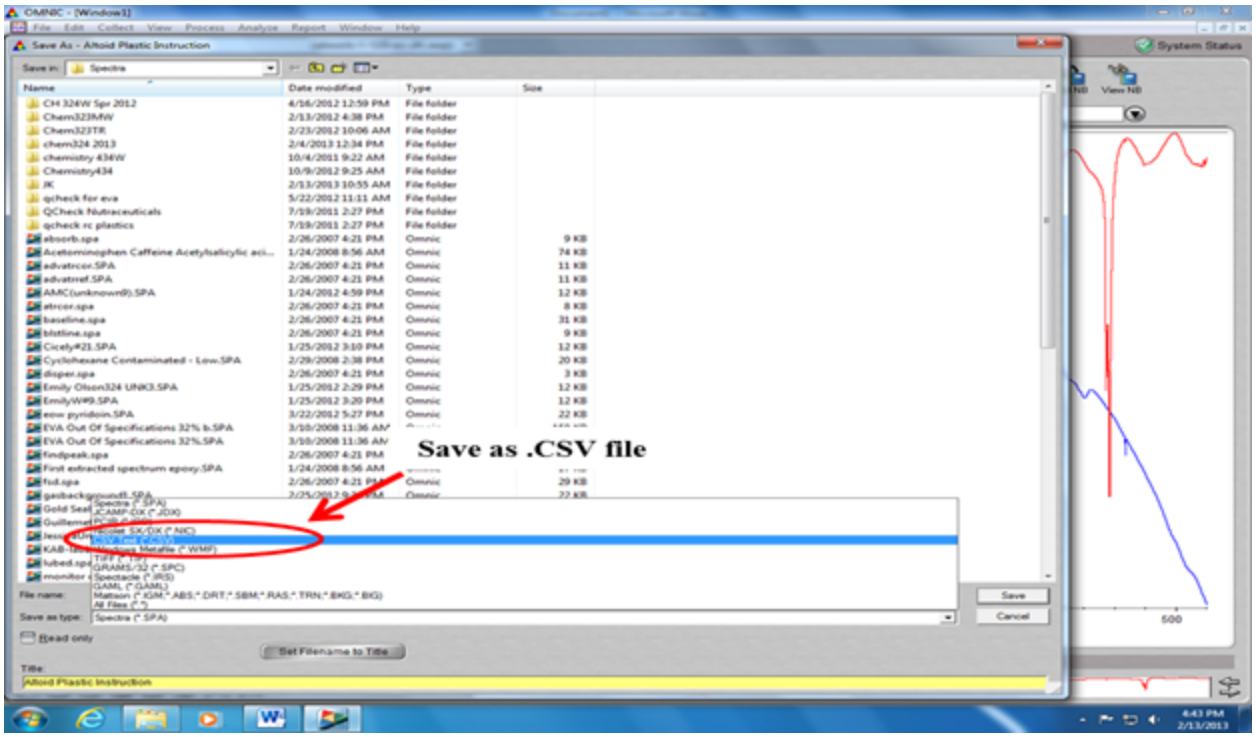
The best match will appear onscreen. Click View Match List to see other close matches.



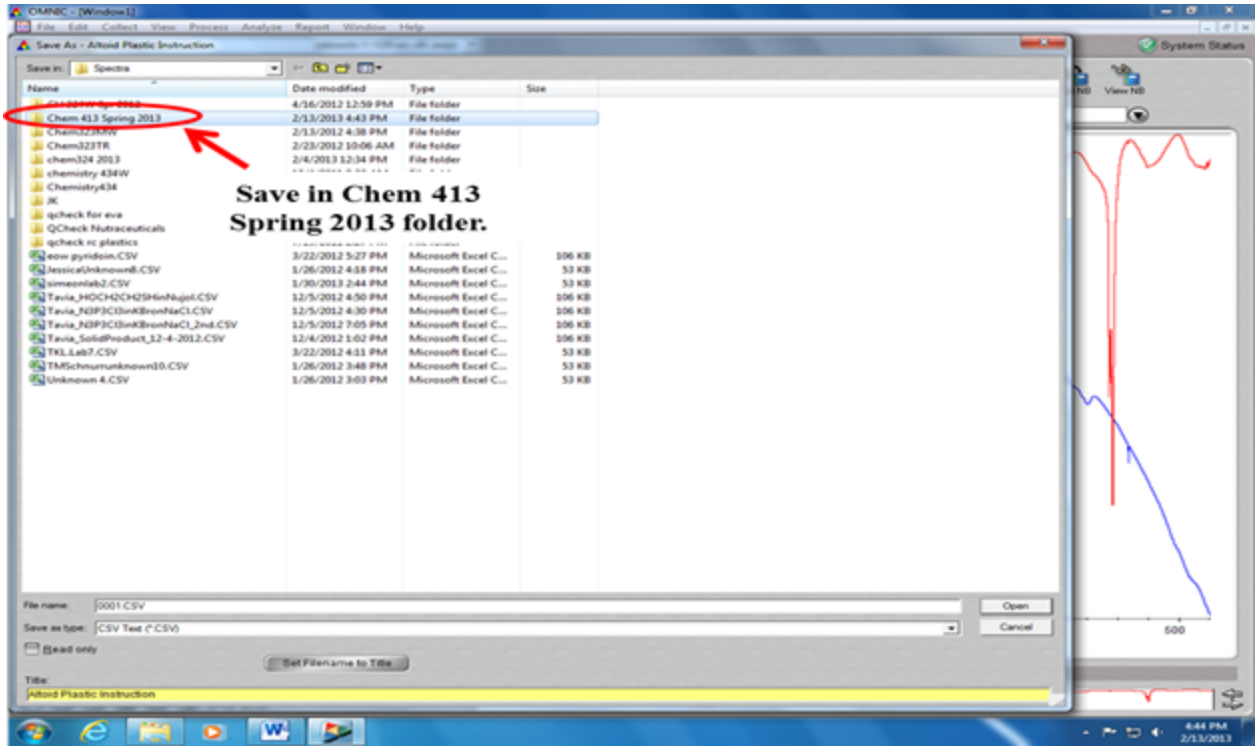
Be sure to record all Match List data. When you are done, click Close in the bottom right.



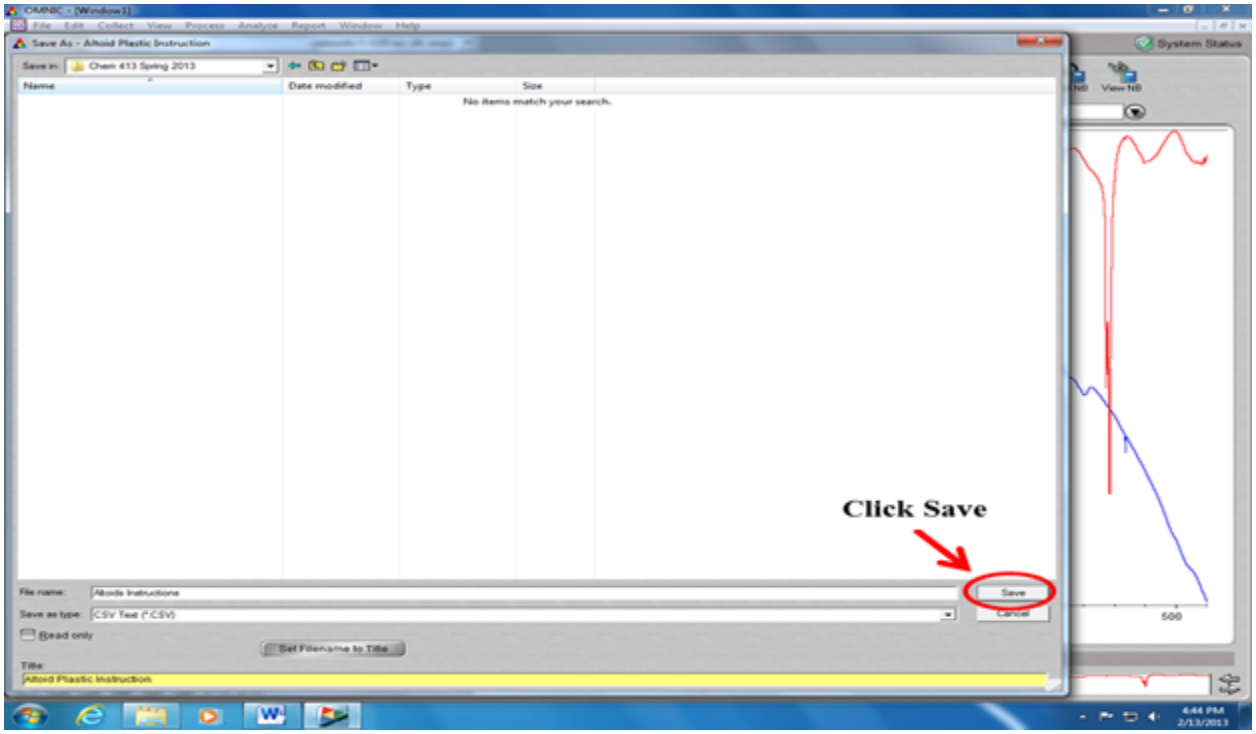
To save your spectrum, open the File menu and click on Save As.
Be sure to save your spectrum as a .CSV file so that you can open it in Excel later on.



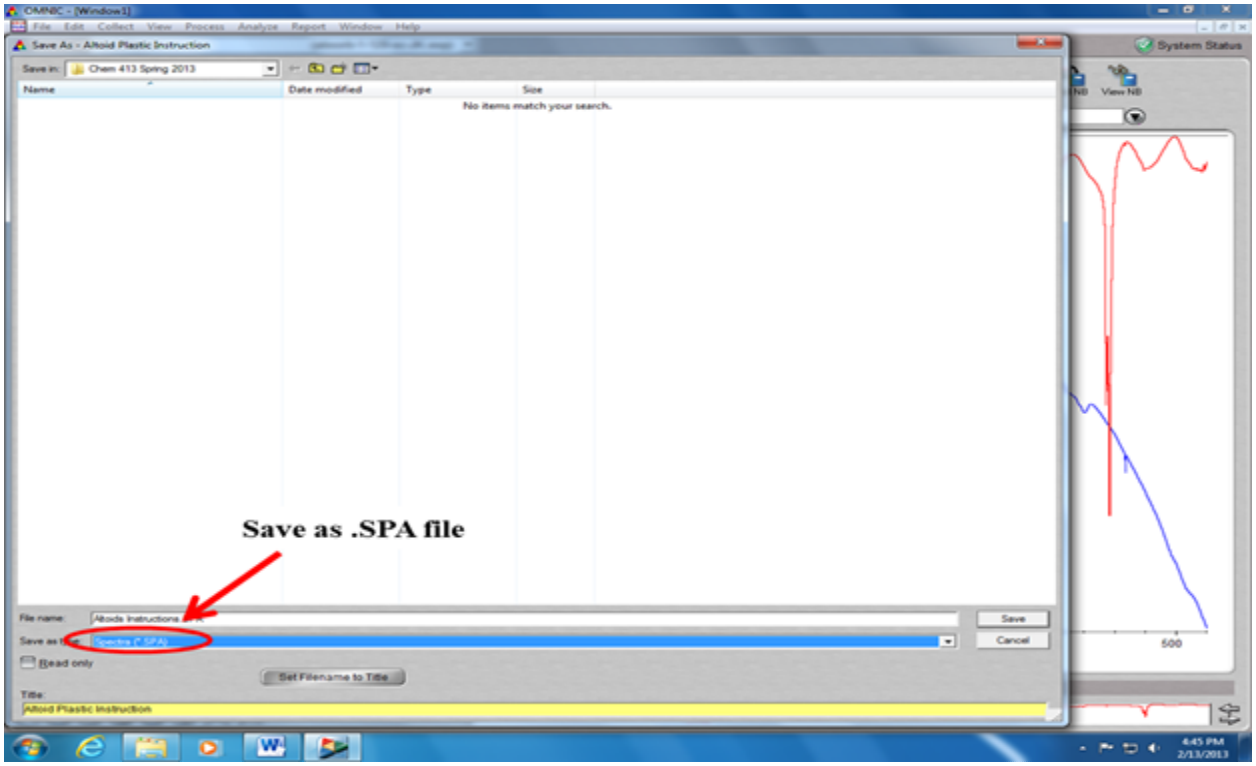
Open the Chem 413 Spring 2013 folder.



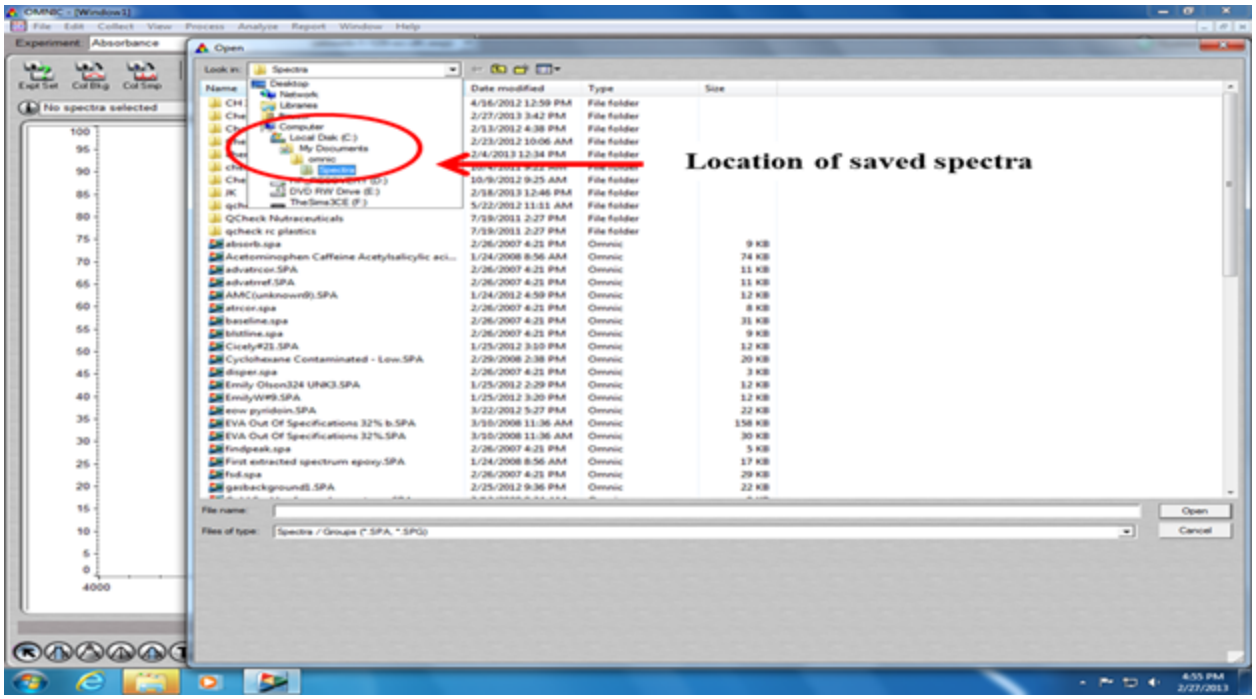
Save your spectrum.



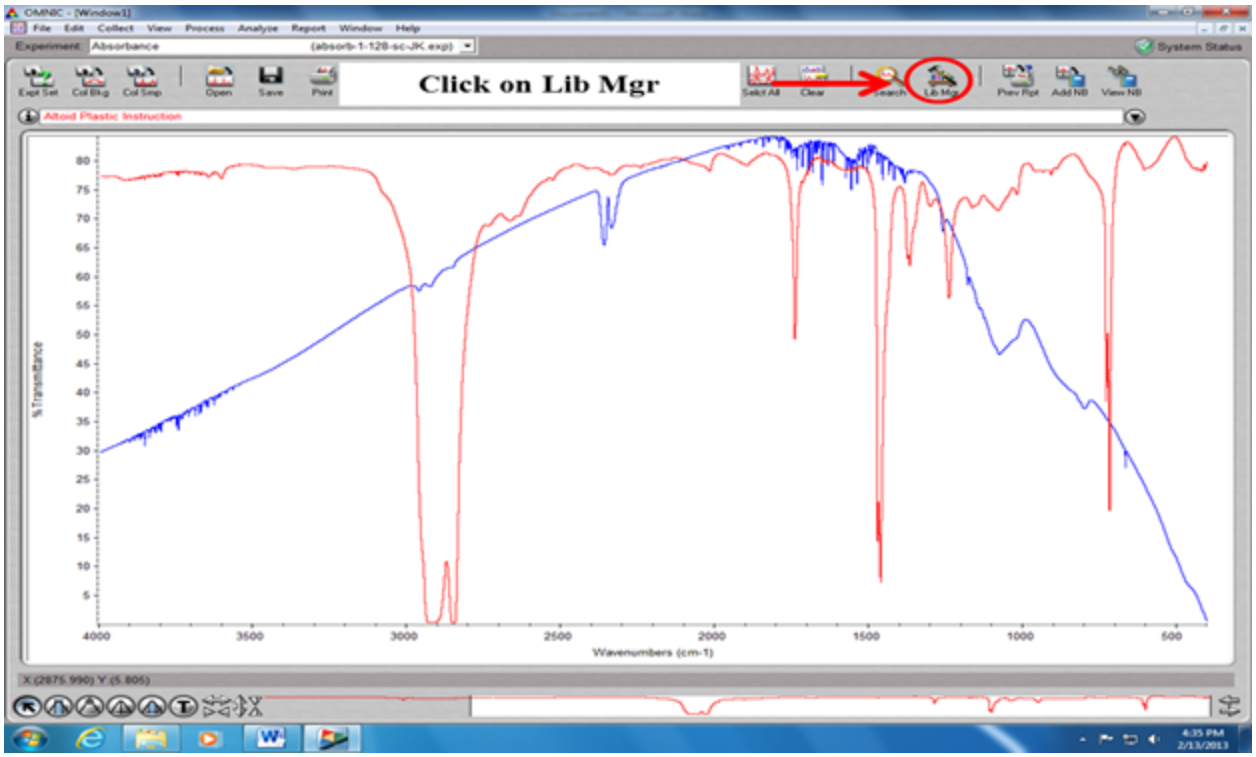
Repeat the steps to save your spectrum, this time saving as a .SPA file.
This will allow you to view the spectrum in the OMNIC software later on.



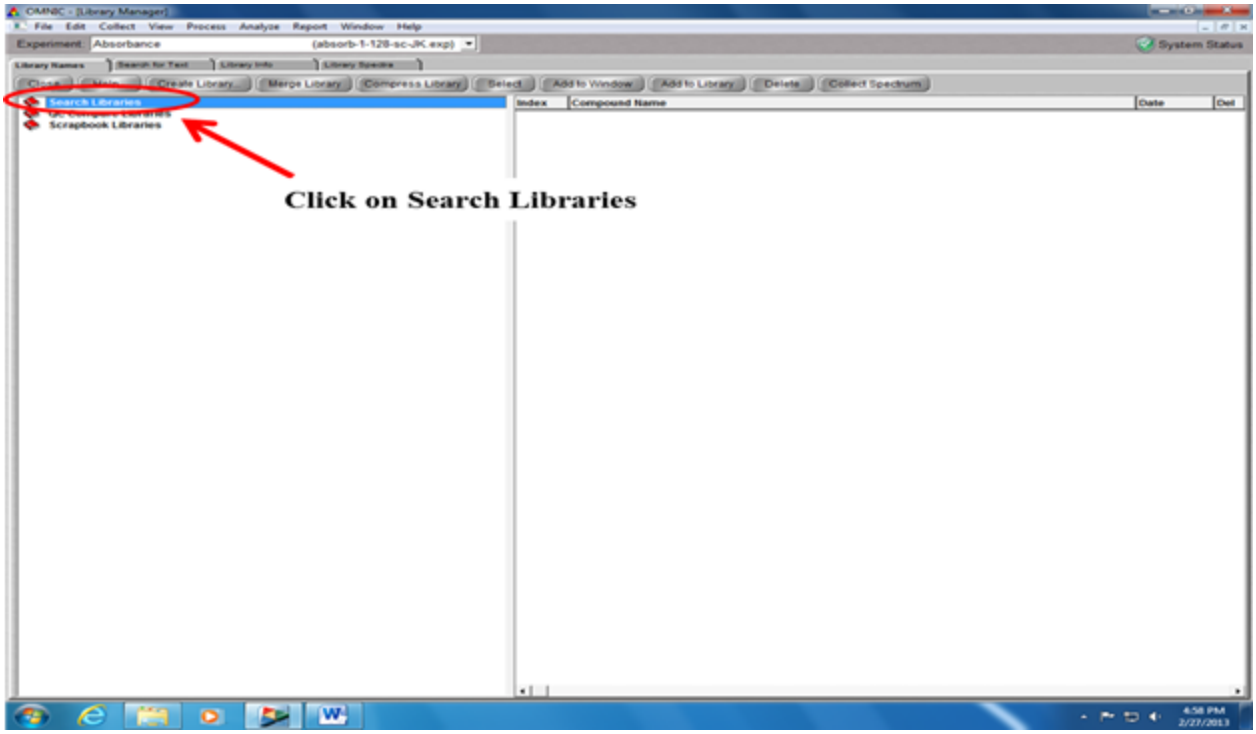
If you need to transfer saved spectra to a USB or upload them to an e-mail, spectra can be found in Computer → Local Disk (C:) → My Documents → omnic → Spectra



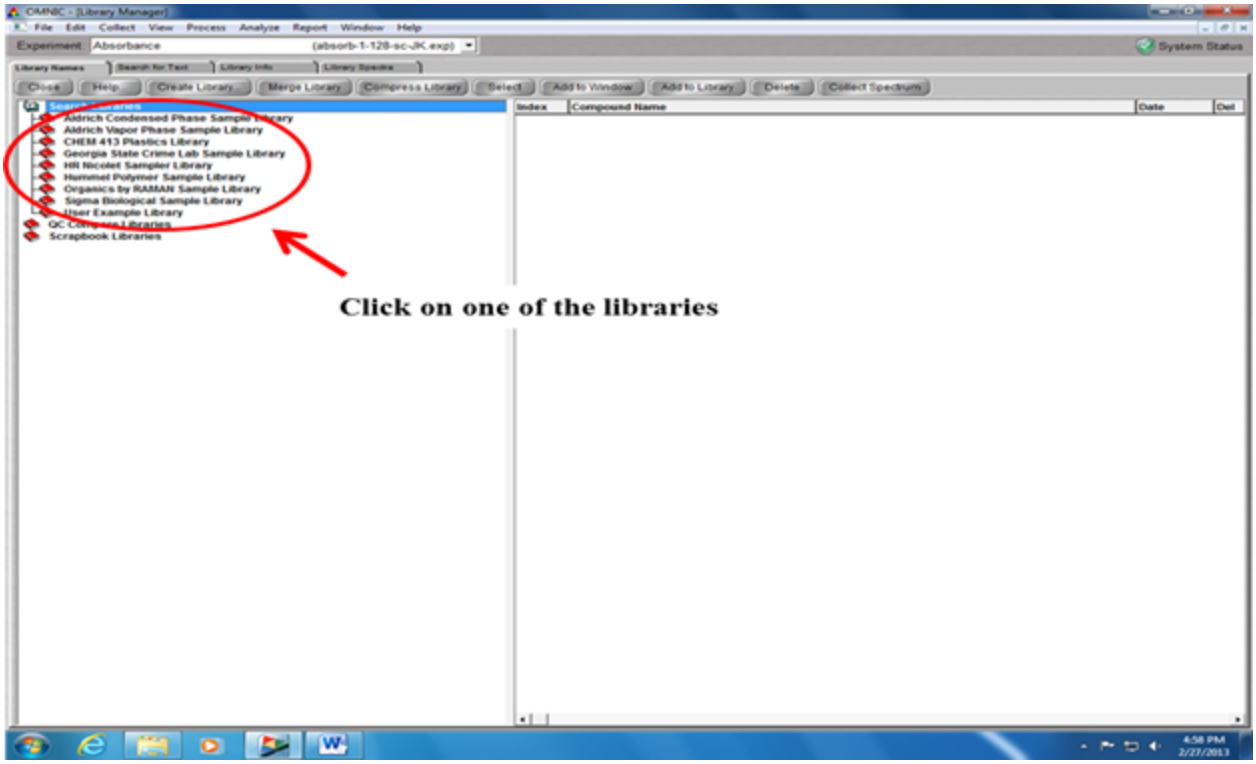
You cannot export a library spectrum to Excel, so it's best to open it up and get a screenshot instead. Begin by clicking Lib Mgr.



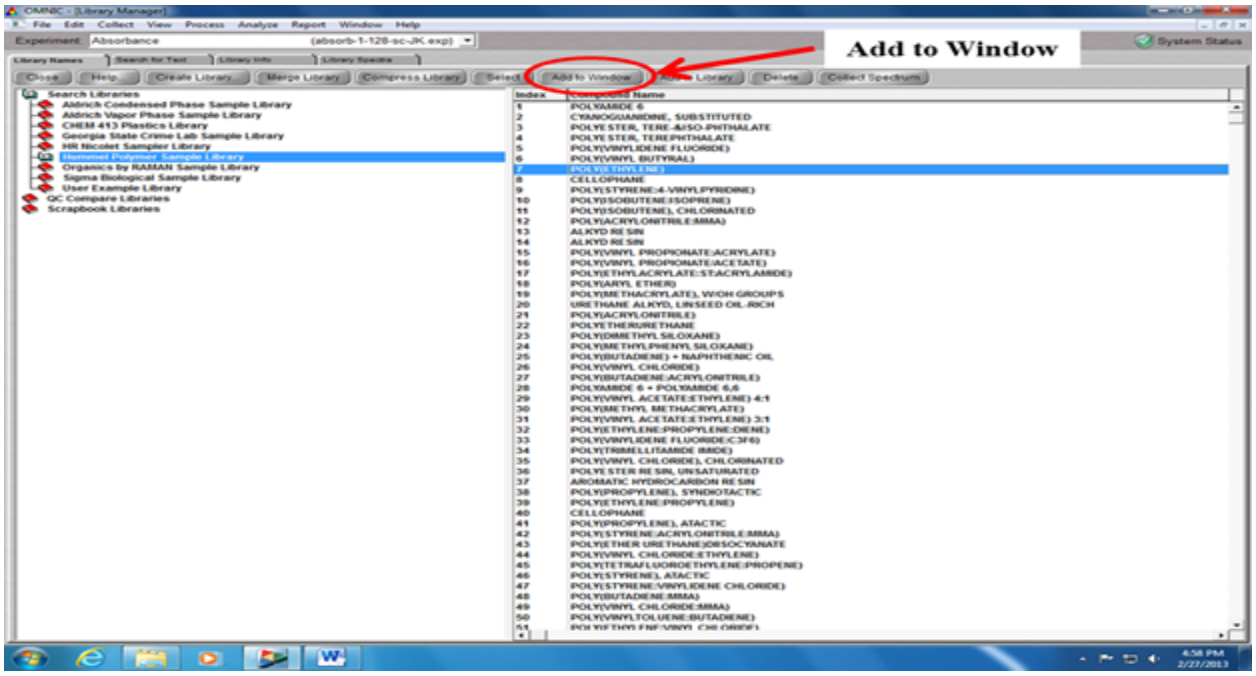
Click on Search Libraries.



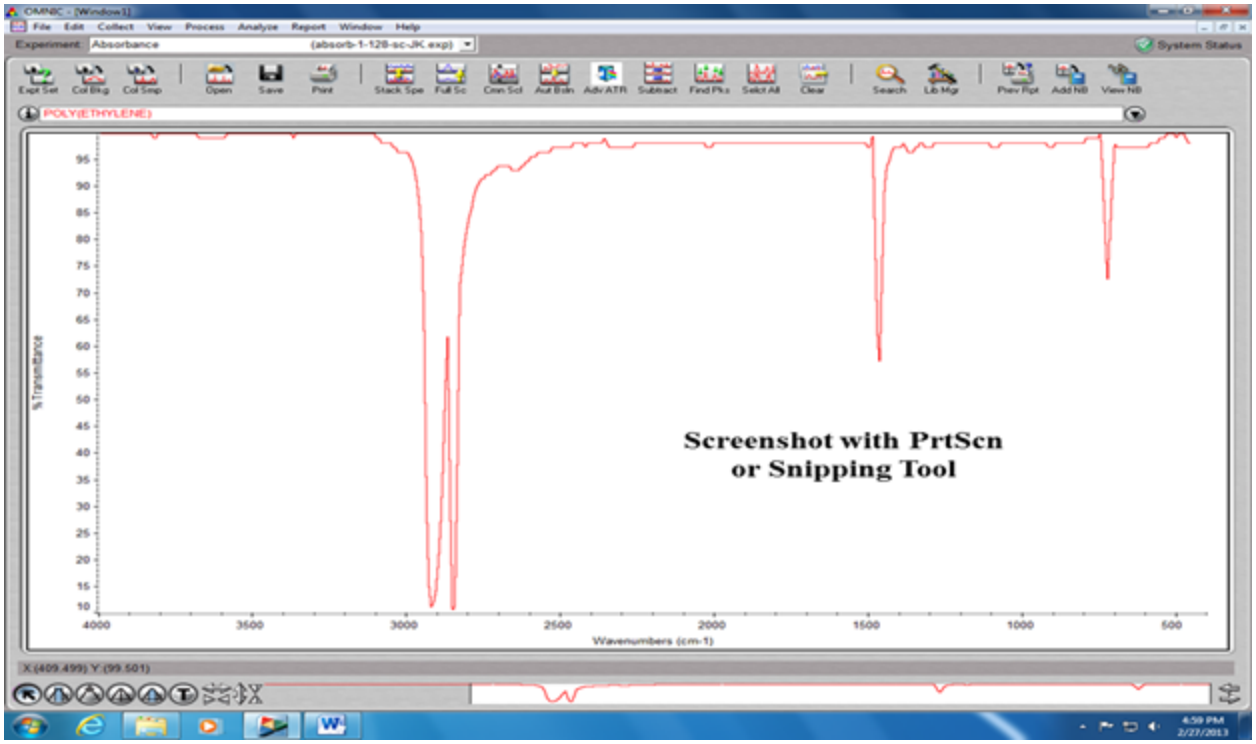
Click on the library which contains one of your matched spectra.



Find your matched spectrum and click Add to Window.
When the pop-up box appears, choose Add to Window1, then click OK.

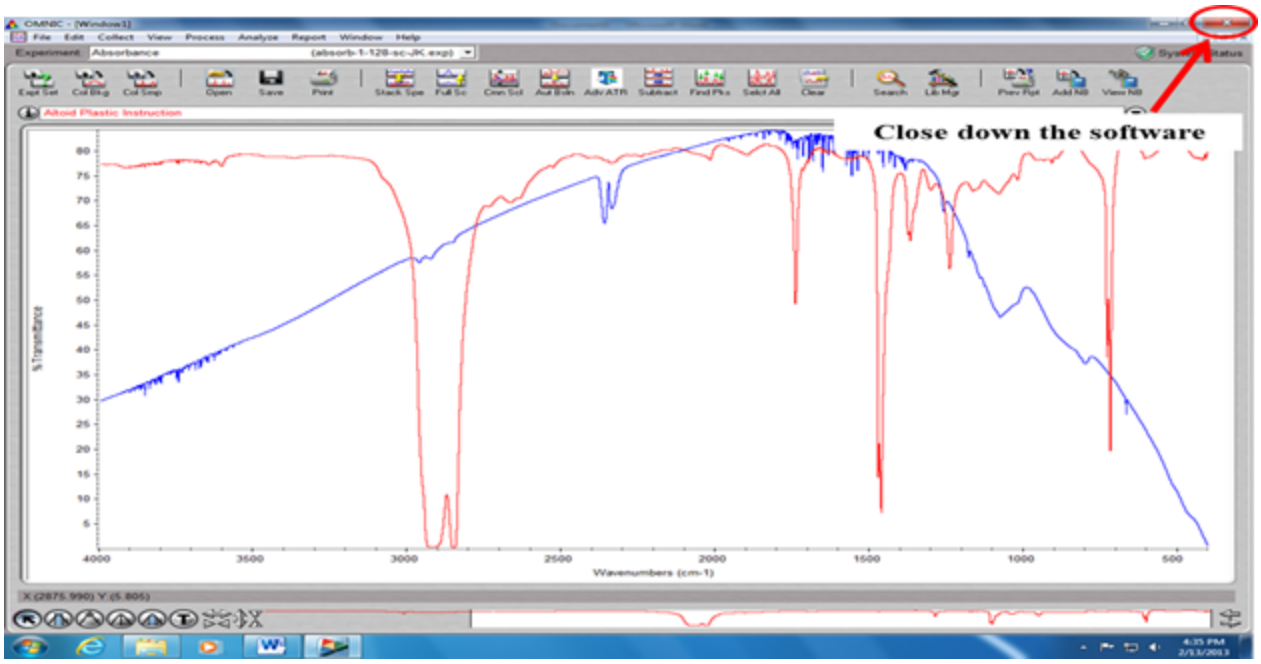


With the library spectrum displayed, screenshot it with the PrtScn button (upper-right of the keyboard) and then paste into a Microsoft Word document. Alternatively, use the Snipping Tool in the Start Menu to capture an image of the spectrum.



Instrument Shutdown

When sample collection is complete and your spectra are saved, close down the software.



The instrument is normally left on after use.

Troubleshooting

Ideally, the absorbance for a sample should be less than 1 AU. However, if a sample spectrum has absorbance greater than this, the sample is too thick and needs to be thinned out.[5]

If a spectrum contains CO_2 peaks that interfere with the data, a new background should be collected.[5]

If a spectrum has too much noise, increase the number of scans or reduce the resolution.[5]

Instrument maintenance

Maintenance tasks for this instrument begin with running diagnostic tests on the instrument components whenever the instrument is not performing properly.[5] Additionally, the spectrometer should be aligned once a week.[5] Once this has been done, accessory components can be aligned as well.[5] Finally, weekly performance tests should be run to track the long-term performance of the spectrometer.[5]

References

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