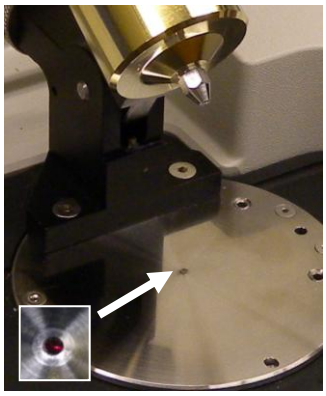
Acquisition of Infrared Spectra on the Nicolet iS10 FTIR Spectrometer

The Thermo-Nicolet iS10 is a research-grade Fourier transform infrared spectrometer. It has both transmission and attenuated total reflectance (ATR) accessories. It can provide data in both transmission and absorbance modes using a standard transmission accessor and also has an attenuated total reflectance (ATR) accessory which can be used for acquiring spectra of liquids and solids for a variety of applications. For training, see Dr. Richard Fitch (Rm. S35E, 237-2244), Dr. Richard Kjonaas (Rm S51A, Phone 237-2237) or Dr. Laurence Rosenhein (Rm. S35M, 237-2243). ***It is imperative that you be properly trained before using this instrument. Improper use of the accessories, particularly the ATR, can result in damage to the instrument.***



The attenuated total reflectance (ATR) accessory for the IR is shown at right. It can handle both solid and liquid samples. It consists of a diamond/ZnSe crystal in the center of the plate (see arrow) through which an IR beam is focused from underneath, bouncing off the crystal face (internal reflectance). A sample applied to the face of a crystal can be examined because the IR beam that is bounced through the crystal penetrates a few microns past the surface of the crystal and into the sample, an effect called quantum tunneling. Thus an IR spectrum can be acquired for a sample, provided it is intimate contact. For liquids this is not a problem, but for solids, a press is used to increase contact.

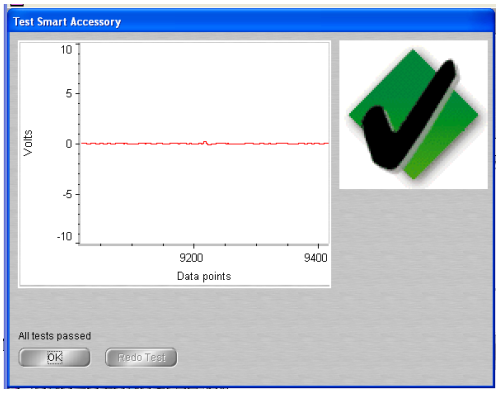
The transmission accessory is a standard window that is typically used for supported samples. The slots are present to support sample holder cards, which can be polymer films for calibration, liquid samples in salt plate sandwiches, KBr pellets for solids, and gas cells.

Changing Accessories. The accessories can be swapped easily for different

applications as shown below. Rotate the locking mechanism on the top of the instrument just behind the accessory and *gently* lift it out of the instrument holding the crossbar in the front and the ATR press (use both hands). *If it does not lift easily, check the locking handle to see if it is completely out of the way.* Place the ATR accessory on the shelf above the instrument and pick up the transmission accessory which should be sitting next to it. Then place the transmission accessory in the instrument. *Be sure to handle the accessories with care, they are very expensive to repair or replace.*

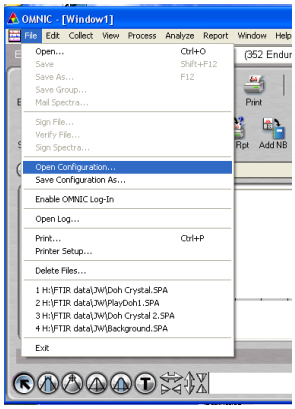


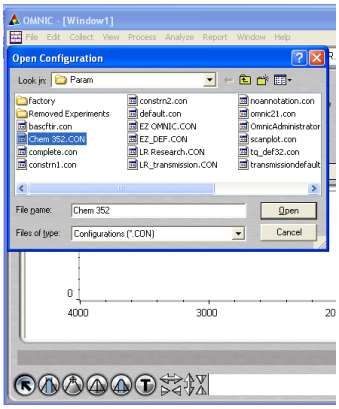
The accessories can be swapped whether the instrument is on or off. If the instrument is on, it will automatically detect the accessory as shown below.

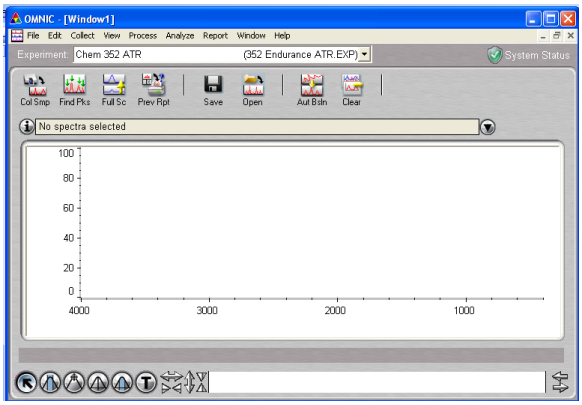


Open and configure OMNIC

OMNIC is the program that operates the FTIR and processes data. Often it will already be running during laboratory class periods. However, it is generally to be turned off after use to save wear and tear on the instrument. If the program is not already open and set up, click on the Omnic icon on the desktop. The program will come up and the instrument will do a self test. The the accessory that is installed may do a self test as shown above (**Changing Accessories**). Once this is complete, the program window will open a default configuration for the instrument as shown below (left). You may use this configuration but it may have too many options for student use in courses. For Chemistry 351L/352L a predefined configuration has been set up. You may set this or another predefined configuration by selecting *File – Open Configuration* (middle). Once selected, the configuration will reorganize the display as shown (right).

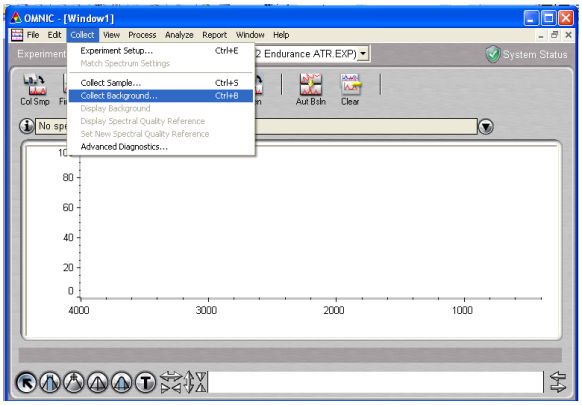


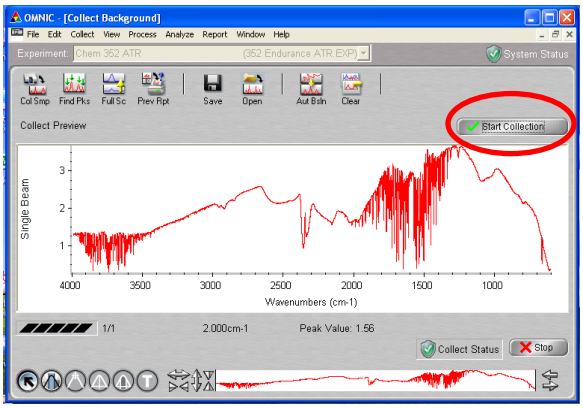




Acquire a Background Scan

Before a sample can be acquired, a background scan must be obtained. Water vapor and carbon dioxide in the air will produce interfering bands which must be subtracted from the spectrum. First be sure there is no sample in the beam and the ATR crystal and press are clean if you are using that accessory (Note: Look closely at the crystal, as leftover films from solids can be tenacious). To obtain a background spectrum, select *Collect – Collect Background* as shown below. The instrument will begin collecting a background spectrum. Observe the bands, which should look similar to that below. If other bands are noted, they may be due to vapor, contamination of the ATR crystal or salt plate if you are using one. Try cleaning with solvent if needed and observe again. There are often vapors present, particularly if many people have been cleaning the ATR crystal with acetone or isopropanol.



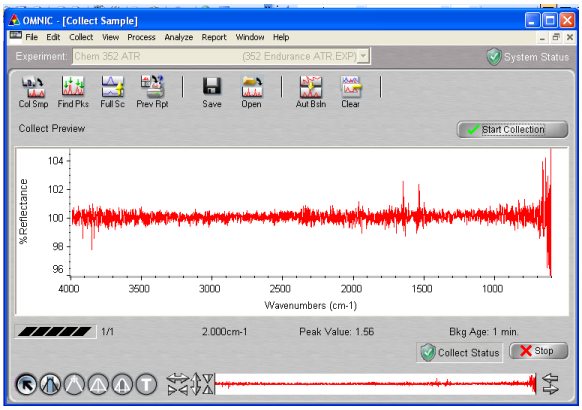


Once you are satisfied that the background is OK, click on Start Collection (circled). A background will be collected and stored. A background is typically good for an hour or two. If you are unsure how long it has been since you acquired your background, you can always collect a new one by repeating the steps above.

Collecting a Sample Spectrum

Once you have a background prepared, it is simplest to collect your sample immediately. To do this , select Collect – Collect Sample from the menu above. The instrument will ask for a spectrum title. For research samples, the notebook number and a short description of the sample are usually best. For class samples, your name and the name of the sample are usually best. Click OK and the instrument will begin collecting a spectrum in scout scan mode. This mode is

helpful to observe your spectrum prior to collection, saving time that can be wasted if the sample preparation is inadequate. The appearance should initially be quite flat as shown below at right.

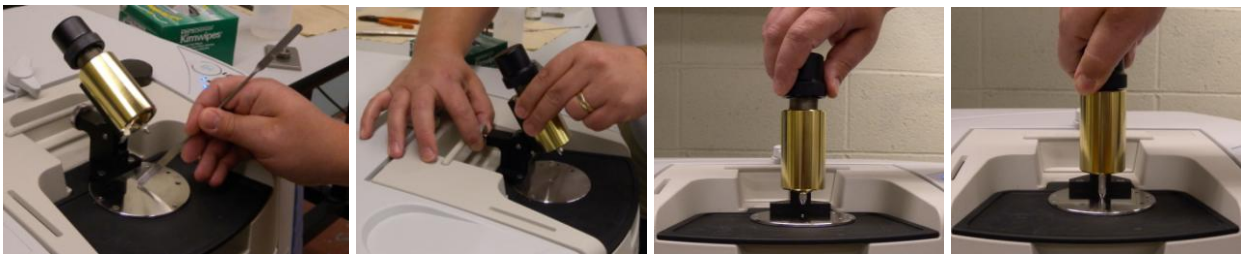


When you have your sample prepared correctly, a proper spectrum will be evident (see below)

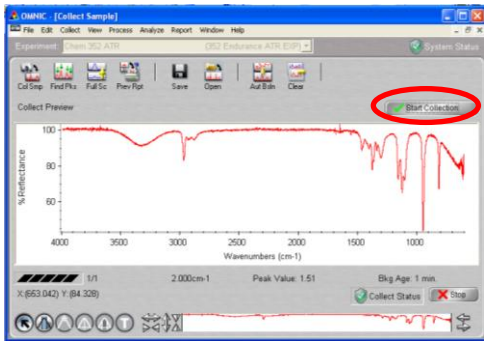
Preparing your sample

Most samples will be analyzed using the ATR accessory as it is convenient for both liquid and solid samples. The transmission accessory can also be used.

Soft Solids. If the sample is a soft inorganic or organic compound place a crystal or tiny amount of powder directly in the center of the diamond crystal in the center of the ATR plate as shown below. Too much powder is not a problem but make sure you clean it up. The IR beam is only 0.1 mm in diameter focused on the center of the diamond. A single crystal of material is quite adequate to obtain a spectrum. Even objects such as a hair or piece of paper can be analyzed. However, you must apply pressure to the sample in order to see it. Lower the press of the ATR and turn the dial until you hear it click as shown below.

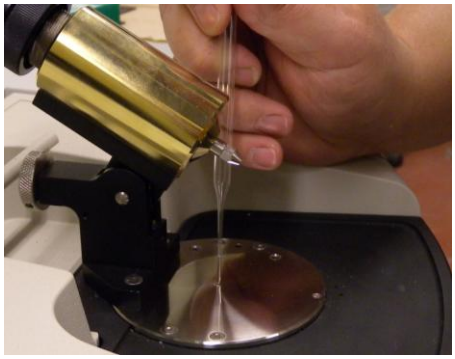


At this point you should see a nice spectrum (right) instead of the flat background. Click on the *Start Collection* button to begin acquisition.



Hard solids. If the solid is a hard inorganic solid, you should powder it first before applying it to the crystal. Although the crystal is made of diamond, it is a laminated diamond wafer over zinc selenide. If enough pressure is applied with a hard material such as sand or glass, it could break and is extraordinarily expensive (~$2,000).

Place the ground powder in the center of the crystal, lower the press of the ATR and turn the dial until you hear it click as shown above. If you do not see a spectrum, either your sample does not absorb in the IR range, or is still too coarse to be seen. Grind the sample further and try again. If you are certain it should absorb IR, ask Dr. Fitch or Dr. Rosenhein to help you.



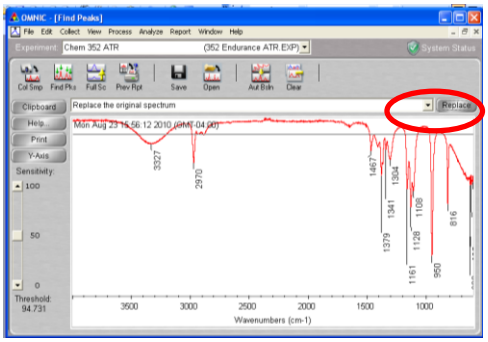
Heavy (nonvolatile) liquids. For a heavy liquid sample apply a tiny droplet of liquid directly to the crystal using the open end of a Pasteur pipette. No pressure is necessary. Simply dip the tip of a pipet (no bulb) into the liquid to be sampled and touch the tip to the crystal until you see a droplet. You should immediately see the acquisition show a nice spectrum instead of the flat background.

Volatile liquids. If a volatile liquid is applied as above, it may evaporate before a spectrum can be acquired. To see this, place a drop of ether or dichloromethane on the crystal. You will notice a spectrum can be seen for several seconds, but then goes away as the solvent evaporates. If your sample is volatile, apply a drop of the liquid to the crystal from a Pasteur pipette and immediately lower the press as shown above. Turn the dial until it clicks and the sample will be trapped. At this point will only evaporate slowly from the edges. A spectrum should easily be acquired before the sample evaporates.

Gases. Gases must be analyzed with the dedicated gas cell using the transmission accessory. If the transmission accessory is not installed, remove the ATR accessory as shown above. If the transmission accessory is already installed, simply acquire a background as described above (**Acquire a Background Scan**). Then collect the gas sample as you would for a solid or liquid as described above.

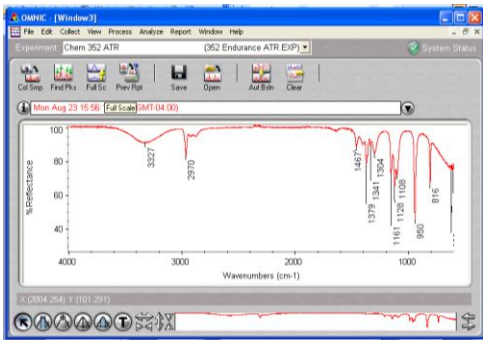
Peak Picking

Once a spectrum has been acquired, you can pick peaks by selecting Analyze – Find Peaks. The peak threshold is indicated by a horizontal line and can be adjusted by clicking on the spectrum. The sensitivity scale on the left can be adjusted to change the number of peaks picked. There is a compromise between selecting noisy peaks and broad peaks. Manual peak picking is not convenient with this software. When finished click on the *Replace* button on the right.



Printing

To print the spectrum, first be sure the spectrum and peak labels are within the window. If they are cut off at the bottom, click on the Full Scale (*Full Sc*) icon in the window. This will give you an appropriate printout of your spectrum. Custom reports can also be set up using the Preview Report (*Prev Rpt*) icon. The spectrum



should print on the networked printer which is on the bench immediately behind you. If printing does not begin, check the printer to be sure it has paper and does not need to be reset.

Save your spectrum

To save your data, simply select *File – Save* or the *Save* icon in the window. Typically data are saved on the departmental server as shown below. Select your course or lab folder as shown below. Again, an appropriate filename will be descriptive with either the notebook number in the appropriate lab folder or your name and experiment in the appropriate course folder.