Optical Lab Room 1122
Nexus 670 and Magna 560
Instructions
Feb. 26, 2013

To get set up to use the FTIR systems, first contact Scott at extension 51806 e-mail staylor7@umd.edu to make appointment for training/orientation (less than 1 hour).

You will be provided with a user name, usually your research group PI's name.

A RFID card will be programmed for you to log on the computer. This is only used for either of the two Nexus 670 systems, not the Magna 560.

Place the RFID card on the red square, the RFID card should automatically enter the user name and password in the logon field

Please write your name, PI, and time on the instrument in the Logbook. There is a scheduler to reserve the instruments:

https://ssl.chem.umd.edu/WebApplications/sac-scheduler

Please notify Scott Taylor of problems or if you have questions call 301-405-1806, staylor7@umd.edu.

The Nexus 670 should always be on. Check LEDS on the top left of the instrument, all should be on, with scan light blinking.

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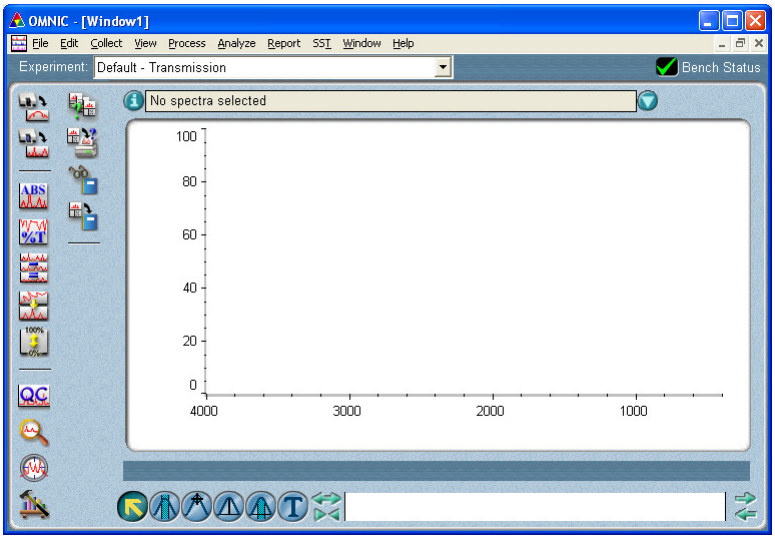
Click on either icon for "Omnic ESP". At this point you can also use the Help Menu for further information in addition to these instructions. See "Getting Started" in the help menu.

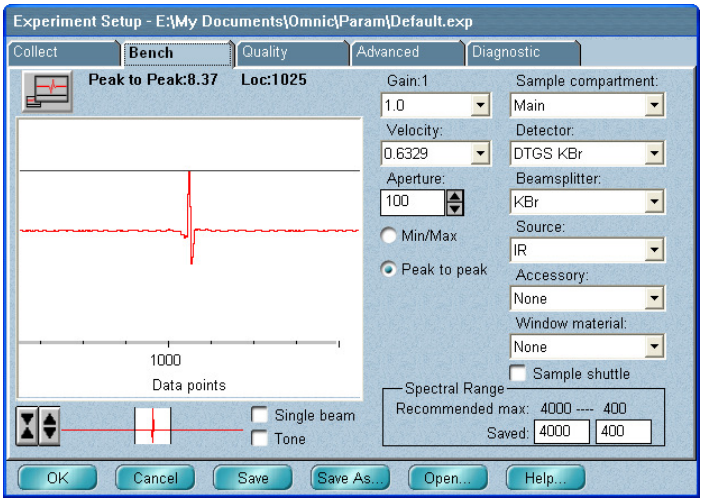
After Omnic opens, look for a green check mark and the words Bench Status in the top right corner. This indicates that the interferometer and the computer are

connected. If there is no icon there, there is no communication between the computer and interferometer. Sometimes there is a few minutes delay while the program is initializing. Note that you still have to check the interferogram as follows.



VERY IMPORTANT!!
Go to Collect, Experiment Setup, Bench, to observe the interferogram. In the middle of the display is a characteristic signal maximum. Check to see if the interferogram signal is within limits, (min 2, max 9.8) if it is a flat line go to Collect, Diagnostics and click on Reset Bench. With a gain of 1 (set by going to the Bench Tab in Experiment Setup) check the p-p voltage-it should be greater than 8 volts. This is with no accessories and a clear path in the sample compartment. Also be sure the aperture is set to 100, normal for resolution 4. A smaller resolution will automatically set the aperture smaller. This means the interferogram peak-to-peak voltage will be smaller.

It is very important to check the experiment setup bench and collect tabs to see if the peak to peak level and parameters are correct. Check the aperture to see if it is 100%, unless you are using a resolution smaller than 4, someone may have change the experiment previously. Also, it is important not to overload the detector with too much signal, which will happen if using the MCT detector option. Be sure to see Scott before attempting this. There is more information about this later.

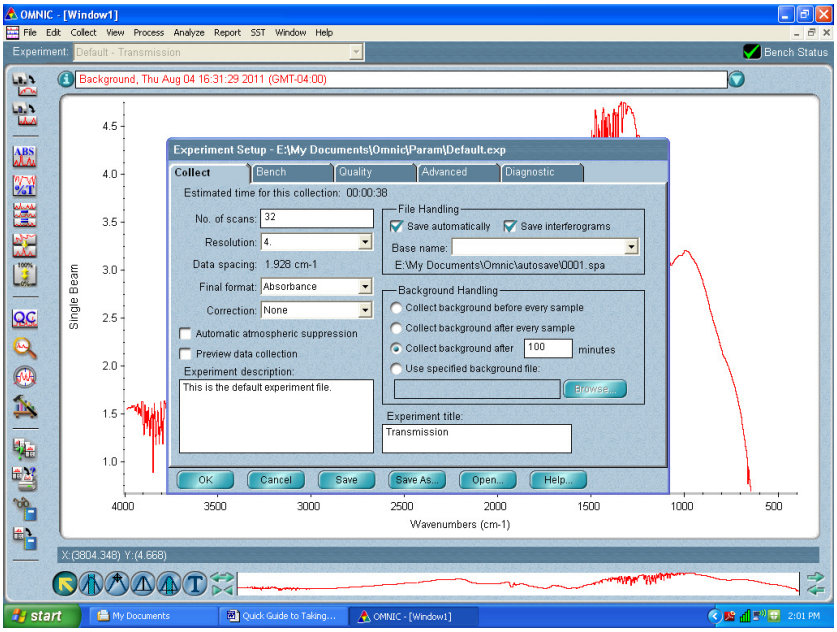


Interferogram Display

Also there is a drop-down window where another detector can be selected. The room temperature detector is DTGS KBr. This is the default detector and normally used.

Normally, the transmission holder should be in the sample chamber. If you use another sample module, such as the ATR module, please carefully clean it and put it back in the cabinet. CHECK TO SEE THE SAMPLE CHAMBER DOOR IS CLOSED WHEN YOU LEAVE THE LAB for more than an hour. (The ATR module must be removed or it will not close.) The sample chamber is purged in addition to the instrument, and helps preserve the windows. If you accidentally leave the door on the chamber open several days, it shortens the life of the windows unnecessarily.

Also, the ATR module is extremely expensive and shouldn't be stored outside of its box and cabinet.

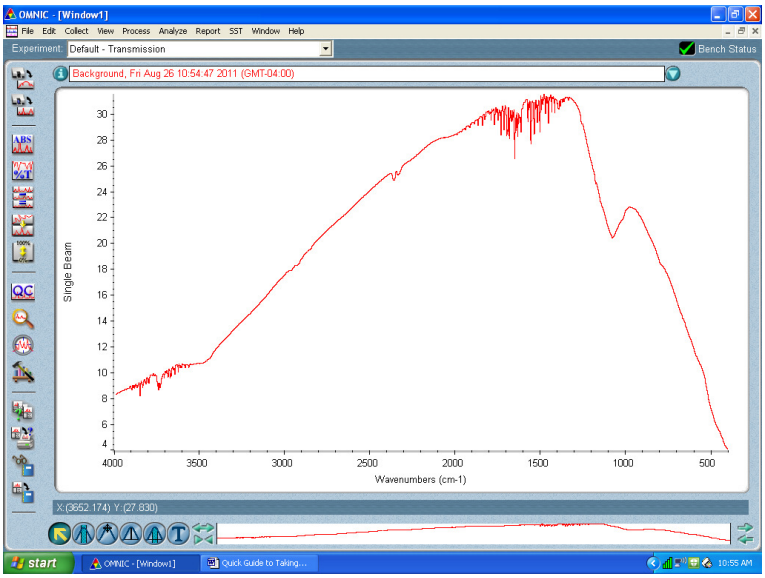


Go to Collect, Experimental setup to select number of scans, default 32, resolution default 4, absorbance or transmission, correction for CO2 and others, and other options.

It is usually set up for the transmission experiment.

Background:

Collect background first with no sample. This will give a characteristic background in transmission mode with no sample as shown below. This shows the instrument operating normally.
Before collecting data, create a background with the solvent, pure KBr pellet or blank KBr or NaCl window so this will be subtracted from sample response. Insert the blank KBr pellet, KBr or NaCl window in the sample holder. Choose Collect Background from the Collect Menu, or click on the Collect Background button on the toolbar.



When the collection is finished, you can save the background to a window if you want it available later for a background handling option, or simply close the Collect Background window. The spectrum is not saved then, but remains the current background. This step eliminates the background characteristics of the spectrometer so the peaks in the final spectrum are due solely to the sample.

Collecting Sample:

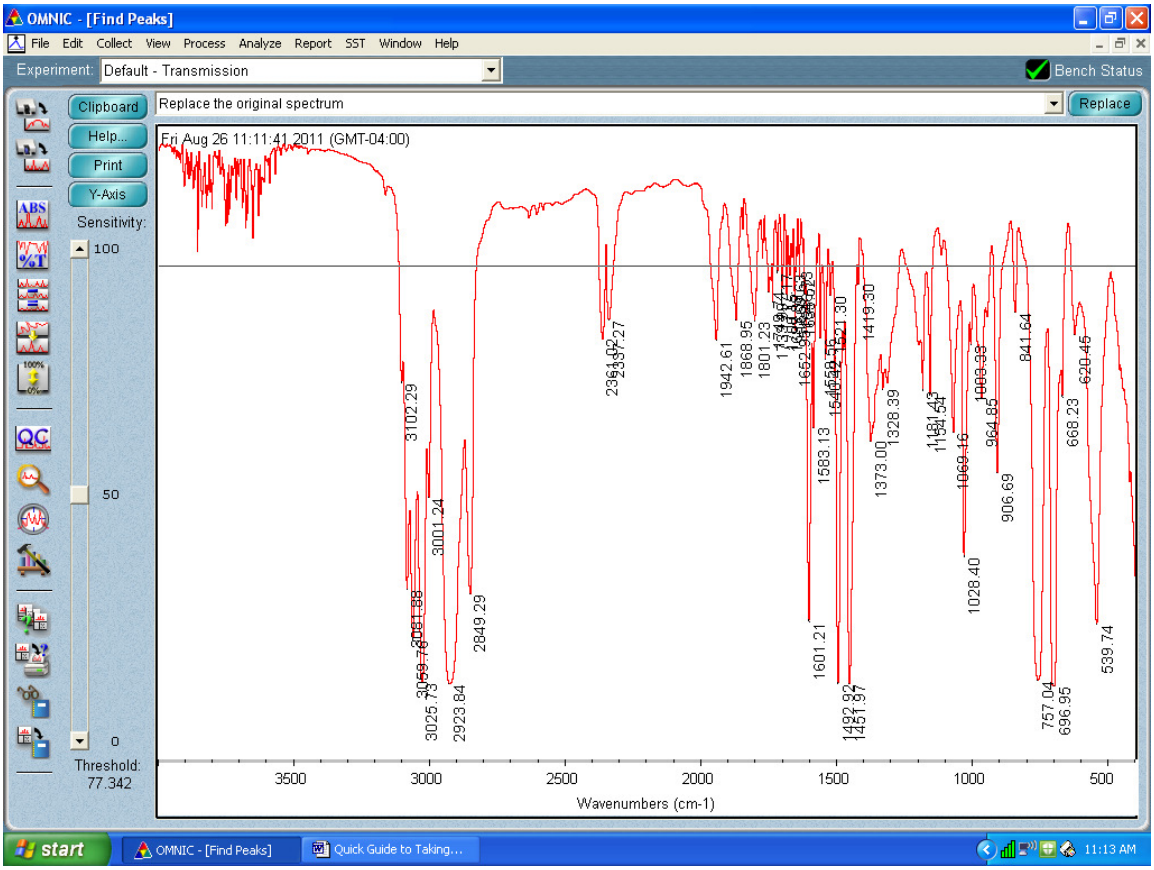
The sample chamber holder holds standard transmission sample cards and liquid sample cells.

Install your sample in the light path, taking care not to spill anything inside the sample compartment. This can be a liquid sample cell, a KBr or NaCl window. The red laser spot will serve to locate the measurement path, it comes from the right side of the sample compartment.

Avoid looking directly or at reflections from this laser and the invisible infrared beam.

Use Collect Sample in the Collect menu to collect the spectrum of a sample, or click on the Collect Background button on the toolbar. There is a progress indicator bar on the lower left hand side of the screen. The spectrum of the sample will be ratioed against the single-beam spectrum of the background.

The result below is a polystyrene film. For a film, the background is air or purge gas.



Edit Menu

This allows you to change the size of the annotation numbers when printing,

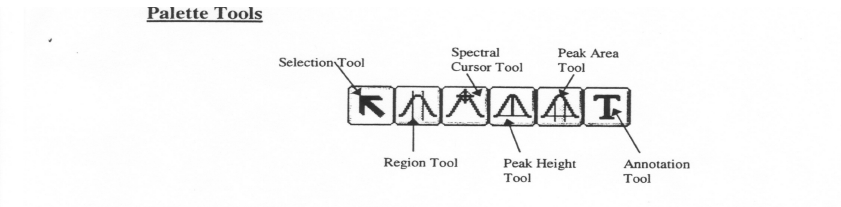
Collect Menu

This can bring up “experiment setup”, with ability to change number of scans and resolution. Final format can be absorbance or transmission. There is a box to check “save interferogram” to have the ability to process the spectrum later. Corrections that can be made later include one for ATR wavelength dependence of attenuated total reflection. Background handling determines when and how often background is taken. Also you can use a saved background file.

Analyze Menu

Annotating the Peaks:
Click on the top toolbar, then Find Peaks. Click on the line where you want to peaks to be annotated. On the left side there is a slider marked “Threshold” to select minimum threshold for peak selection. Click on Replace button on top right before printing.

. The palette tool labeled T will select individual peaks.



Selection Tool: Display specific regions, zoom in and out, grab and move spectra, select single or multiple spectra, define (x,y) coordinates. This is the default tool when you start the program.

Region Tool: Selects X-axis start/stop frequency, use for manipulations such as library searches, blank/straight lines, noise calculations, uncorrected area calculations, (all are discussed in further detail below)

Spectral Cursor Tool: Reports value of data points (x.y); used for spectral comparisons; if you have a log file running, the data points will be recorded in the log.

Peak Height Tool: Calculates peak intensities, allows user-defined baseline, calculates height-to-zero as well as relative measurements, stores measurements in log file.

Peak Area Tool: Integrates the area under a peak, allows user-defined baselines, calculates area-to-zero as well as relative measurements, stores measurements in log file.

Annotation Tool: Manual labeling of spectrum, labels are stored with the spectrum (highlight to delete), style and font is adjustable (Display setup and Options, see below).

SHIFT KEY + Click of mouse will pinpoint the maximum for Spectral Cursor, Peak Height, and Annotation tools. Automatically finds the point of greatest intensity near the cursor.

RIGHT Mouse **Click** will bring up an information box for whatever the cursor is resting on. The box will always have a "Discussion" button and sometimes a "How To" button to help you with the features of the object.

File Menu

This allows you to save in different data formats.

Accepted Formats	Extensions
OMNIC Spectra	*.SPA
OMNIC Group	*.SPG
JCAMP-DX	*.JDX
PCIR	*.IRD
Nicolet SX/DX	*NIC or *.SPC
Grams 386	*.GLD
Peak Solve	*.0??
Mattson	*.IGM, *.ABS, *.DRT, *.SBM, *.RAS
Windows Metafile	*.WMF
TIF	*TIF
Comma Separated Value	*.CSV

Also, if you go to file, print, cute pdf will convert to pd

"Save Group" allows you to save several spectra within the same file.

Sampling Methods

There are many sampling methods in transmission mode, including pressing KBr pellets, coating a KBr or CaF2 window, or using a liquid sample cell. Sometimes these methods must be used if the ATR method doesn't provide the information you need.

The ATR Module

The ATR module is very expensive and many precautions should be taken before using it. The ATR module allows quick sampling of solids or liquids without preparation like the transmission methods. The tradeoff is that the infrared beam only penetrates the sample in microns and the method has less sensitivity than transmission measurements.

The ATR , or Attenuated Total Reflectance module has a very small window which the sample can contact. Infrared light is reflected by a mirror to a conical zinc selenide lens with a diamond coating on it. USE GREAT CAUTION when touching this surface, do not hit it with the steel anvil. Turn the knob

counterclockwise to retract the anvil before you move it into place. Don't touch the surface directly with the steel anvil. The steel anvil is only intended to press solid or powder samples against the window, not used in background or liquid measurements.

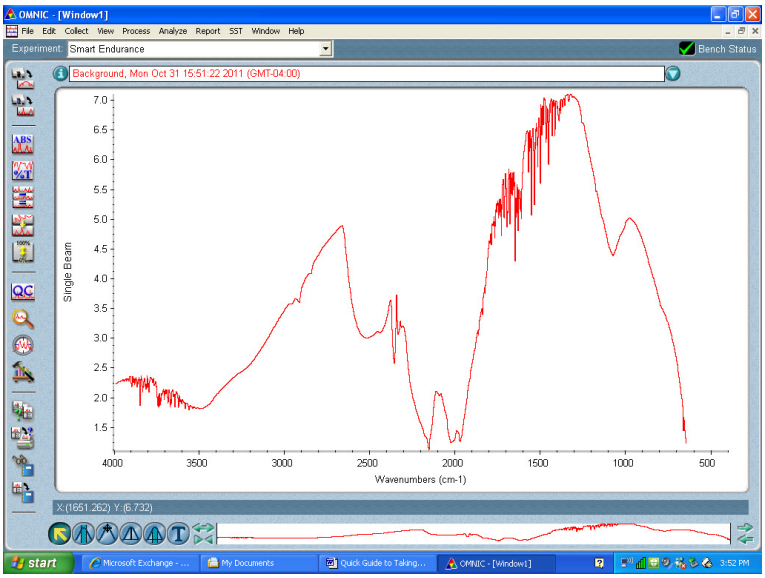
Take background measurements with the cell window clean. Don't lower the anvil down because air is the reference we use. Also, there is a risk of damaging the window surface. Use auto gain for the gain setting.

A small amount of liquid to cover the cell is enough when taking a sample. Don't allow liquid to run down the plate, carefully wipe with Kim wipe to stop spillage.

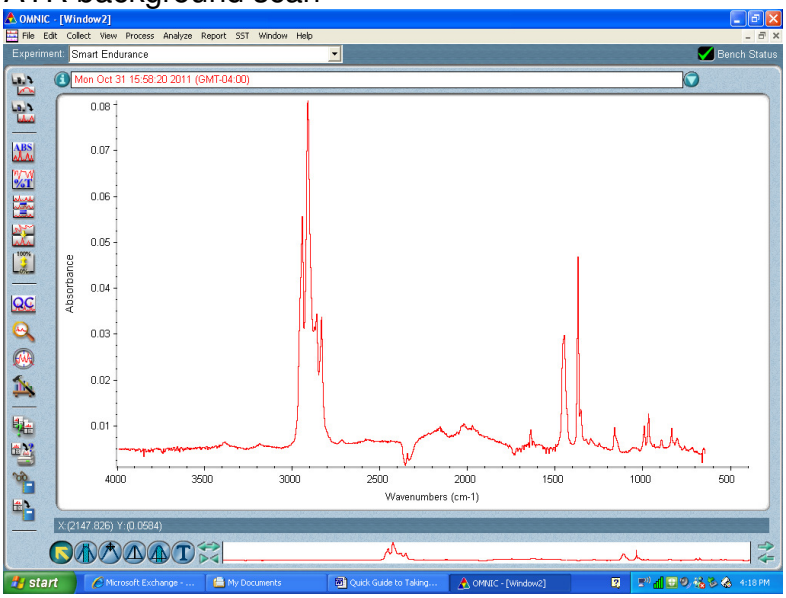
When taking powder samples, use a small amount to cover the cell. Turn the knob counterclockwise to retract the anvil. Pull back on the smaller knob to release the arm and lock it down. Then turn the knob clockwise until the anvil presses down on the sample material. The knob will click when there is enough torque. Then collect the sample. Carefully wipe powder up into the kimwipe with ethyl alcohol, do not drop powder over the edge of the plate or anywhere else.

The ATR method saves time, but the hardware is very expensive and requires neatness and care. Please see pictures below to show sample accessory removal, but contact Scott Taylor X 51806 before you do it the first time.

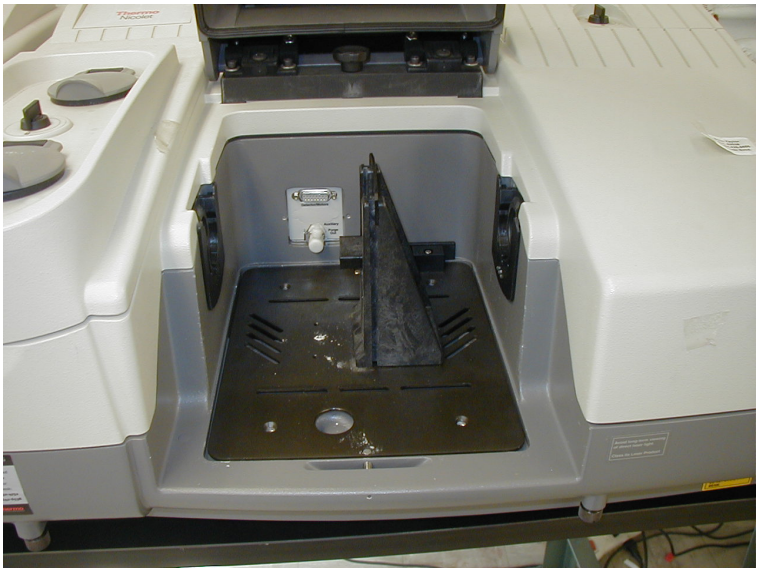
Always return the instrument to the transmission module and close and latch the cover when finished.



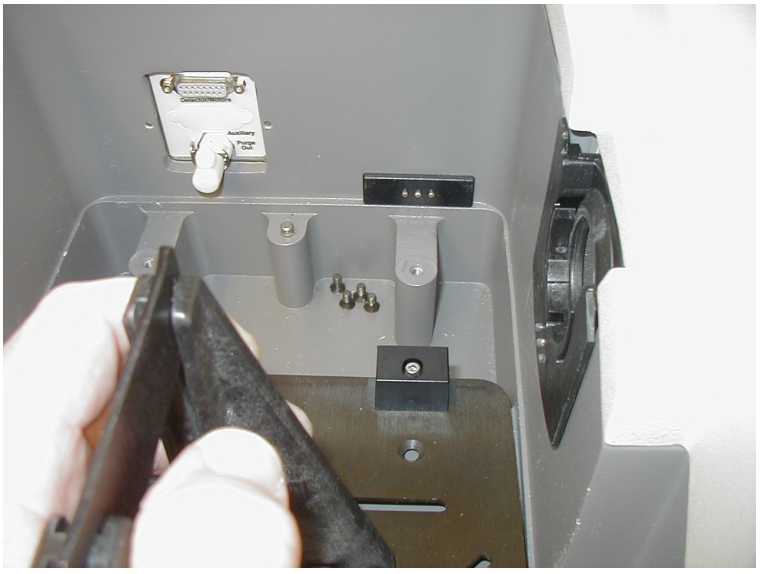
ATR background scan



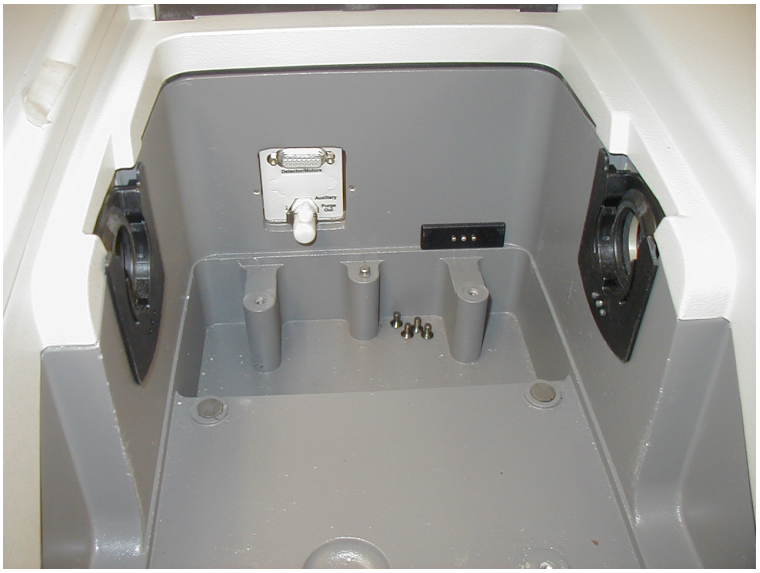
ATR sample of 3 layers of plastic wrapping material



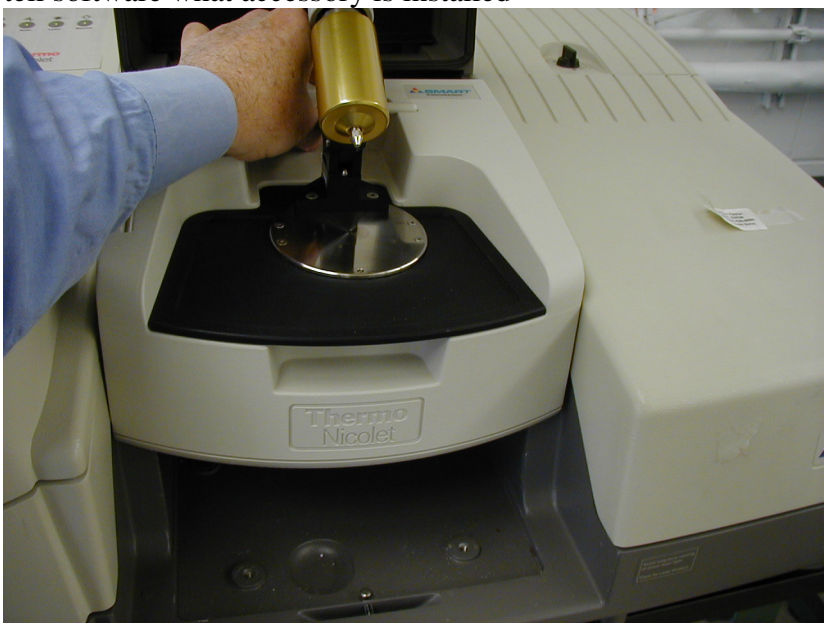
Top picture-Transmission sample accessory installed.



Bottom picture-removing Transmission sample accessory. To remove, pull up to clear pins. Two magnets hold the plate so that screws are not needed. Carefully pull back to remove.



Top picture, empty sample compartment- Note rows of contacts on rear and side, these tell software what accessory is installed



. A self test program runs after the accessory is seated. Lower very carefully, rocking it slightly to get it to seat properly.

ATR Module with plastic sample:

Top picture, ATR fully seated in the sample compartment.
Bottom picture, anvil with plastic film sample pressed onto sample cell window.←←

The MCT/A Detector

For much greater sensitivity and smaller resolution settings use the MCT/A detector. This is used with transmission measurements. Also, most of the time you will need to use the pure nitrogen instead of the regular purge gas to reduce the water and CO2 background, especially if it is in the region of interest. The MCT detector is selected in Experiment setup, Bench tab in the drop down window. Please see Scott Taylor before using this detector, as it must be cooled by liquid nitrogen. Fill with liquid nitrogen as described in Omnic Help.

Because the MCT detector is so much more sensitive, the interferogram signal will be much larger than the 9 volts peak to peak limit. There are energy reducing screens which fit in the left side window of the sample chamber. Use the B screen, usually. Otherwise, you will need to use a smaller aperture setting to limit this to the correct level. Also the resolution setting will be automatically reduced to a smaller number than the usual 4, (see experiment setup, Collect) as small as 0.5 cm-1

Screen installed in left side of cample compartment

Liquid nitrogen filling instructions from Omnic Help:

WARNING!
Liquid nitrogen is extremely cold and therefore potentially hazardous. Avoid Contact with Skin. Wear protective clothing and follow standard laboratory practices to prevent injury.
Time-40 minutes or less
Tools:
Protective clothing
Funnel with Styrofoam base-should be near one of the instruments
A one-liter, metal vacuum bottle
Liquid nitrogen

1. Open the dewar cover on the instrument (the one closest to the back) and remove the plastic stopper from the detector dewar.
2. Fill the metal vacuum bottle with liquid nitrogen
3. Insert the funnel into the detector dewar
4. Fill the detector dewar with liquid nitrogen. *Fill the detector dewar slowly. Pouring too quickly can cause the funnel to expel liquid nitrogen. Wear protective clothing and follow standard laboratory safety practices to prevent injury.*

Pour the liquid nitrogen into the funnel *slowly*. A small amount of liquid nitrogen spillage is unavoidable and will not harm the instrument, but watch for pooling on the metal surface and stop pouring if this happens.

Fill the funnel and then let it drain completely two or three times. Wait until the vapor plume disappears and then repeat until the dewar is filled.

Your will notice the geyser effect when the dewar is full and the detector is cooled.