

## Collecting a FT-IR Spectrum Using the Standard Pike ATR cell.

Here is a quick **outline** for what to do to collect an FT-IR using the standard Pike ATR cell.

1) Click on the Advanced Measurement icon:



2) **Basic Tab:** Check that the correct XPM experiment file is being used. This will usually be the **Tensor27\_ATR.xpm** experiment file. If someone else's experiment file is loaded, reload the **Tensor27\_ATR.xpm** experiment file (or your custom XPM file) by clicking on the Load button.

3) **Basic Tab:** Type in a description of your sample

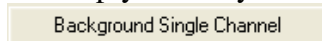
4) **Advanced Tab:** Type in the name for the file that your IR spectrum data will be stored in. Make sure the path to YOUR directory is set.


5) **Advanced Tab:** Check the resolution is set to 4 cm-1, background & sample scans = 16, result spectrum = ATR spectrum.

6) **Optic, Acquisition, and FT Tabs:** Check that these settings are OK (see details later).

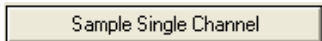
7) **Check Signal:** Check the IR intensity - should be around 4800-5000 counts.


8) **Basic Tab:** Run your background with the cell empty. After you click on the




button the FT-IR will start scanning the background. The progress of the scans is indicated in the bottom middle of the screen: 

9) **Basic Tab:** Add your sample to the crystal (see sampling details under Pike cell)

10) **Basic Tab:** Run your sample spectrum by clicking on the  button.

The progress of the scans is indicated in the bottom middle of the screen: 

Your spectrum (ATM corrected absorbance mode) should then display (usually autoscales, but if not click on the autoscale icon: )

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## Bruker Tensor 27 FT-IR & OPUS Data Collection Program

### Tensor 27 FT-IR Introduction

The departmental FT-IR system is a Bruker Tensor 27 system (purchased 2004) and is located in Choppin 702 (Figure 1). It is equipped with a room temperature DTGS detector, mid-IR source (4000 to 400  $\text{cm}^{-1}$ ), and a KBr beamsplitter. Maximum resolution is 1  $\text{cm}^{-1}$ . This system is free to use. The FT-IR software keeps a log of every user so there is no physical log to fill out. There is an identical Tensor 27 FT-IR system located in Choppin 616 mainly used by the Stanley group for catalytic studies, but available as backup or for extended special FT-IR studies.

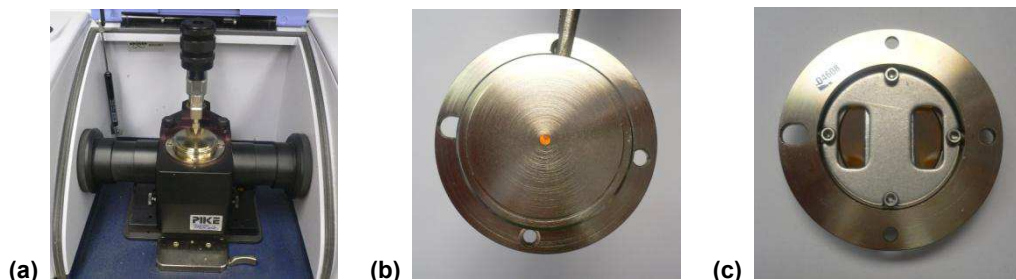
**This is a single-beam instrument so it is very important to generally run a background spectrum first!!** The background will often be the empty cell, but for solution spectra you should run the solvent as the background. This is discussed in the data collection section.

To use the FT-IR in Choppin 702 you must first ask Bob Zinn or Greg O'Dell (Choppin 100A or 100) to set up an account on the computer (Windows XP). They will also setup a username for you to login to the OPUS FT-IR data collection program. See Bob or Greg for any general computer problems you have with this system. **You are responsible for backing up all your data on a regular basis.** Network access instructions are provided towards the end of this document under "Computer Network Access".

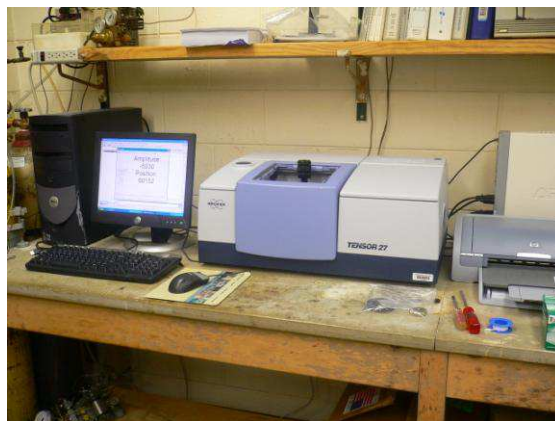
Once you have an account you should either get Prof. George Stanley (Choppin 614, 8-3471, gstanley@lsu.edu) or another student in your group familiar with the FT-IR to check you out. See Prof. Stanley if you have questions about the optical bench, sample cell, or the FT-IR software program (OPUS).

### Sample Cells

The standard sample cell in the FT-IR is a Pike Miracle single-bounce attenuated total reflectance (ATR) cell equipped with a ZnSe single crystal. **ZnSe absorbs strongly below 650  $\text{cm}^{-1}$  and data should not be collected below this wavenumber.** The cell is shown in Figure 2(a), with close-ups of the top of the cell in Fig 2(b) showing the orange color of the ZnSe crystal spot upon which the sample is placed. Fig. 2(c) shows the bottom of the crystal plate with the bulk of the ZnSe crystal that you do not see.



**Figure 2.** (a) Pike Miracle ATR cell. (b) Close-up of the top sample plate with the ZnSe crystal illuminated. The sample should cover the orange crystal surface. (c) bottom of the sample plate showing the rest of the ZnSe crystal that the IR beam travels through.



**Figure 1.** Bruker Tensor 27 FT-IR in Choppin 702.

The ATR beam path for the cell and crystal is shown in Figure 3. The sample is placed directly on the small crystal spot and the arm rotated over and turned down to press the sample down onto the crystal face to get better contact. The IR beam penetrates about 60  $\mu\text{m}$  into the sample. If your sample is extremely strongly absorbing (e.g., carbon nanotubes) you will not get much of an IR spectrum. But this ZnSe ATR cell works well for most “normal” organic and inorganic samples. For extremely strongly absorbing samples a Germanium crystal works better for ATR (we don’t have one), or you make a dilute KBr pellet or a thin film on KBr.



Figure 3. IR beam path for Pike ATR cell.

The sample that is placed on the ATR crystal can be solid, gel, liquid, or solution (non-corrosive, naturally). Since it will be out in the air the sample needs to be oxygen and moisture stable (for air-sensitive samples read on). You only need enough sample to cover the crystal surface and, if a solid, enough for the “crank down arm” to press the sample onto the crystal to have good contact with the crystal surface. The ZnSe is fairly soft, so be careful with “hard” solid samples.

The turning handle has a built-in clutch that slips when enough downward force is applied, so you don’t have to worry about over tightening it. It also swings out of the way at any point above the crystal and has “catches” to guide it into place above the crystal surface or at the 90° away mark. If it gets out of alignment ask Prof. Stanley to readjust it.

Figure 4 shows part of the sequence of events to measure a sample of plastic film (Parafilm). **First, measure a background spectrum with nothing on the crystal** (discussed under the OPUS software section)!! Then place the sample onto the crystal, move the lever arm over so it snaps into place above the crystal, then turn the top handle so the slightly concave stainless steel tip presses the sample onto the crystal face.

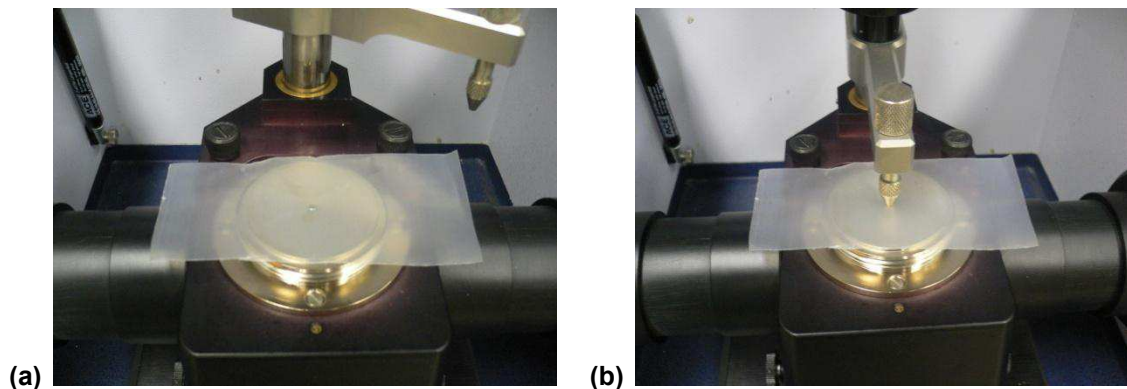


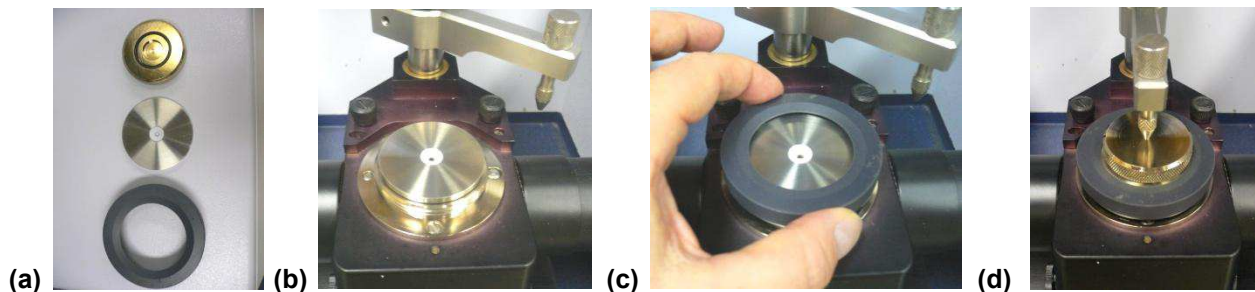
Figure 4. (a) Placing plastic film on crystal surface (way too big, but OK). (b) Sample depressing arm has been rotated over the crystal and turned down till the clutch slipped. Sample IR was then run.

For traditional solid samples, powders work best. Although one could place a large single crystal of a compound directly onto the ZnSe crystal, mineral-like crystals (hard) could definitely scratch up the relatively soft ZnSe sampling crystal. Most organics and transition metal coordination compounds/complexes are softer or have similar hardness to the ZnSe.

Gels can be smeared right onto the crystal. Be especially careful about cleaning up gooey or gel-like samples.

**Liquid or Solution:** A pure non-volatile liquid can be placed directly onto the crystal for a measurement. The sample pushing lever arm has a slight concave inner surface that will trap some liquid against the crystal so it doesn't evaporate quite so quickly, but it is not air-tight. So it is generally good to lower the arm to trap the liquid against the crystal surface.

For solutions or liquids that evaporate more quickly there is a liquid sampling accessory sitting on top of the FT-IR in a zip-lock bag. Figure 5 shows the sequence of photos for using this.



**Figure 5.** (a) Three parts of the liquid sampling accessory. (b) Place disk with Teflon depression onto sample plate, ZnSe crystal should be centered in the middle of the hole. (c) Screw on black outer ring to press the Teflon portion tightly against the crystal rim. (d) **Pure liquid:** run background spectrum before adding your sample, then add sample into the Teflon depression (just a drop), cover with brass plate to minimize evaporation and run your sample spectrum. **Solution:** run background spectrum with your solvent in the Teflon depression (just a drop) after covering with the brass plate, then use a Q-tip or Kimwip to soak up the solvent and give it some time to dry. Then add your sample solution, cover with brass plate to minimize evaporation and run your spectrum. Aqueous solutions are OK. NO strong acids or bases.

Although the Teflon disk makes a pretty good seal against the base plate, there may be a bit of leakage underneath (keep this in mind when cleaning up).

This cell is NOT very sensitive for samples in solution. You need fairly concentrated samples (around 0.2 M) to get a semi-reasonable spectrum. Some dilute sample solution spectra are shown later under the data collection and scan time section – they are not very good. Those of you that experiment with solutions with this Pike single-bounce ATR cell, please provide me feedback and I will include that here.

You can also use this liquid sampling accessory for solids as the conical Teflon section is useful for directing the solid sample onto the crystal. You would not use the brass cap for a solid sample. Instead, crank the arm-press tip down onto the solid to make sure it is pressed firmly against the crystal surface.

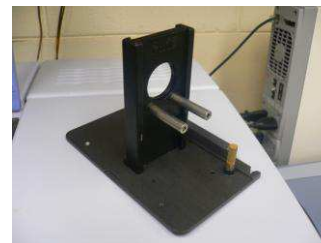
**CLEAN-UP:** It is very important to clean the cell surface well after you are done. There should be a bottle of isopropyl alcohol, kimwips, and Q-tips to the right of the optical bench. **Please refill or replace these from your own labs as they are used up.**

Acetone is OK for cleaning the cell surface BUT it may take the paint off the IR optical bench and eat away at some of the plastics (note damage on computer case!!). **Do NOT FORGET to clean the bottom of the steel lever arm that pushes down on your sample!!** If you use the liquid sampling setup shown in Fig. 5, make sure that you clean the stainless-steel/Teflon disk well.

**Salt Plate/Pellet/IR card Holder:** This is shown in Figure 6 and is sitting on the shelf on top of the FT-IR optical bench. The pellet/plate holder is inserted (Fig. 6, bottom) but can be removed. This can allow you to insert other disposable IR-card type sample holders such as screen, KBr, or polyethylene film (Fig. 7).



**Figure 7.** IR screen (goeey samples), KBr real crystal, and polyethylene sample cards.



**Figure 6. (top)** IR card-salt plate holder and separate pellet holder. **(bottom)** assembled.

To switch cells one just needs remove the existing cell by pressing the cell release lever located at the right front of the base locking plate (Figure 8), tilt the front of the cell up and out of the front locking plate, then remove the cell.

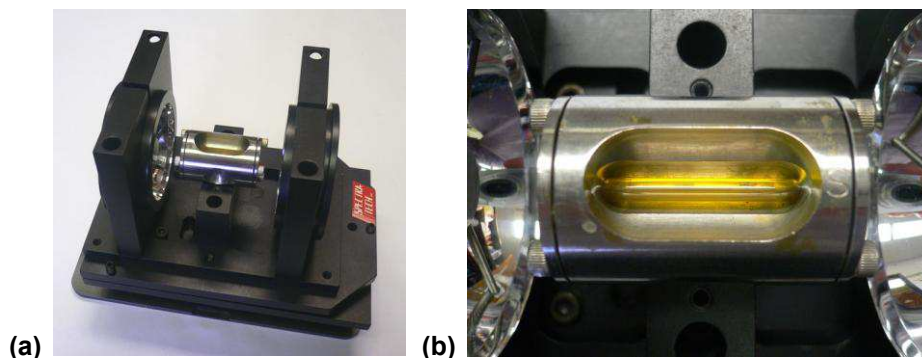
The new cell is inserted with the cell base plate electronics connection pointed to the back of the FT-IR cell locking system. Fit the back portion of the cell in first with the front of the cell tilted up, then press down and lock into place.

Most of the newer cells for the Tensor 27 have base plates with electronics that uniquely identifies the cell to the spectrometer. There is an automatic accessory recognition (AAR) feature that can pre-load reference background spectra for a cell, set data collection parameters, and enable simple sample collection. This AAR feature is not currently activated, but may be in the future. See Prof. Stanley and later sections in OPUS for more information.



**Figure 8.** Cell base plate unlocking lever.

**SpectraTech Circle ATR Solution cell:** This cell is shown in Figure 9 and can be obtained from Prof. Stanley. Although it is an older technology it works well for solutions and small scale reactions. The sample cell has a 4.0 cm long cylindrical ZnSe crystal rod. The IR beam bounces

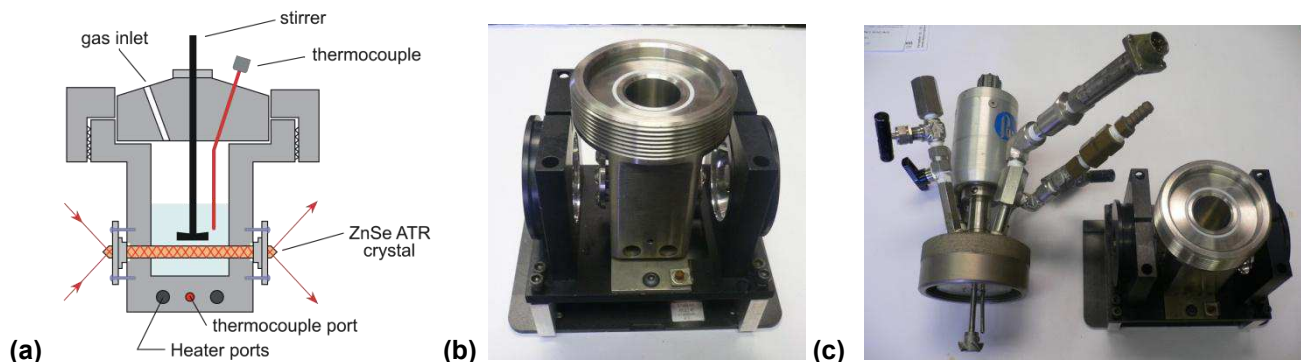


**Figure 9.** (a) SpectraTech Circle micro-ATR solution cell. (b) close-up of removable ATR microcell.

through the rod resulting in multiple sampling points throughout the solution. The solution volume needed is about 0.5 mL. **The sample concentration in solution should be at least 50 mM.** 100-200 mM sample concentrations will give good spectra. There originally was a thin metal plate to cover the top to limit solution evaporation. This cover is missing, but we can rig up some plastic to do the same job.

Due to the longer IR beam pathlength through the crystal rod the more sensitive liquid nitrogen (LN<sub>2</sub>) MCT (mercury-cadmium-telluride) IR detector needs to be used. The MCT detector is stored in Choppin 616 with the other Tensor system and can be obtained from Prof. Stanley. Instructions for using or filling the MCT detector with LN<sub>2</sub> are given in the next section on the FT-IR optical bench.

**SpectraTech High Pressure/Temp Circle ATR Solution Reaction Cell:** This is a 40 mL Parr stainless steel mini-autoclave with a hole drilled through it for a ZnSe ATR crystal rod. It can handle gas pressures up to 1,500 psig (100 atm) and temperatures up to 200°C. A schematic drawing and photos are shown in Figure 10.



**Figure 10.** (a) Schematic drawing of high pressure circle rxn cell. (b) Photo of cell bottom mounted in holder. (c) Photo of cell bottom along with the stirrer head piece, valves, thermocouple, and electronic pressure transducer.

This circle rxn cell is operated by a Parr 4850 autoclave process controller shown in Figure 11, which can control and track the temperature, stirring rate, and the pressure of the autoclave, along with the pressure of an external high pressure gas reservoir. A manual pressure regulator is available with the external gas reservoir to deliver a constant pressure gas or gas mixture to the IR cell. Prof. Stanley's group has used this cell for studying hydroformylation catalysis by mono- and dinuclear rhodium complexes. For their studies that focus



**Figure 11.** Parr controller for the high pressure circle rxn cell.



on the metal-carbonyl band region ( $2200\text{-}1700\text{ cm}^{-1}$ , typically strong absorbances) metal complex concentrations of at least 30-50 mM were needed to get decent spectra of just the metal-carbonyl region.

As with the regular solution circle ATR cell discussed above (very similar ATR technologies), the high sensitivity MCT detector must be used.

For more information on this system, see Prof. Stanley.

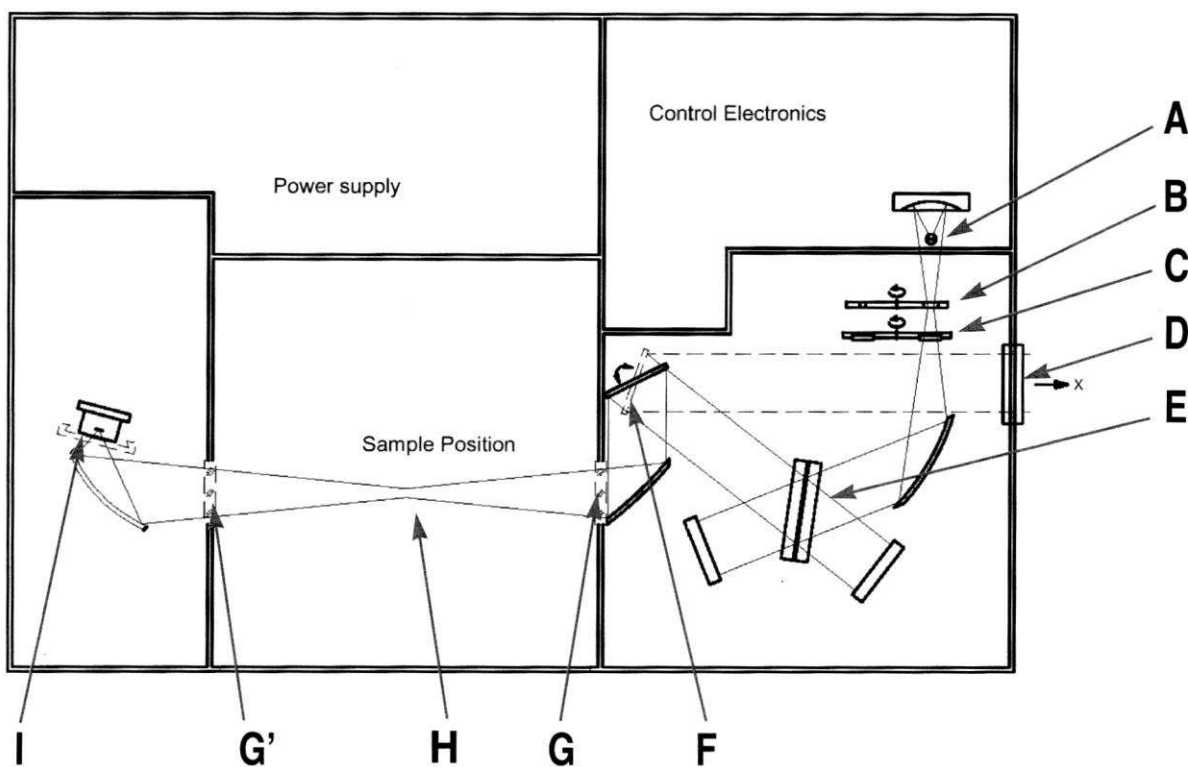
**Air-Sensitive Samples:** Collecting the FT-IR of oxygen or moisture-sensitive samples can be quite a challenge. Air-sensitive solution IR's can be fairly easily collected using the high-pressure circle rxn ATR cell discussed above. A septum replaces the head assembly for ambient solution data.

A nujol mull of an air-sensitive solid sample prepared in a glove box can be put between two KBr plates and into a zip lock bag for transfer out of the glove box and to the FT-IR for use in the KBr plate sample holder. The diffusion of  $\text{O}_2$  or moisture into the center of the mull sandwiched between the two plates is generally slow enough to obtain good FT-IR spectra on the central portion of the plates.

For other air-sensitive sampling methods and special procedures see Prof. Stanley.

## Tensor 27 Optical Bench

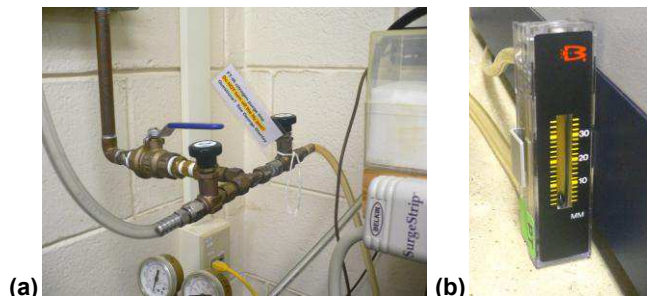
The Tensor 27 FT-IR optical bench has no external controls and is operated completely (aside from the power switch on the back) from the computer system. The optical path of the bench and major components are shown in Figure 12.



**Figure 12.** Schematic of Tensor 27 optical bench, IR beam path, and major components. **A** = mid-IR source, **B** = aperture wheel, **C** = filter wheel, **D** = exit port (not used), **E** = beamsplitter, **F** = switch mirror, **G** = sample compartment window (KBr), **G'** = optional window (not present), **H** = sample holder/cell, **I** = detector (DTGS standard).

The optical bench should be continuously purged with a slow flow of house nitrogen to keep moisture and CO<sub>2</sub> out of the source, interferometer, and detector compartments. **Do NOT turn the N<sub>2</sub> off!!** The inside of the Pike ATR cell is also connected to the N<sub>2</sub> flow to further improve the signal-to-noise. Note that the Pike cell has sleeves that extend out to fit over the source and detector holes. Make sure that these sleeves are flush against each side of the sample compartment to maintain the N<sub>2</sub> atmosphere inside the cell and spectrometer.

The main N<sub>2</sub> shut-off/on valve is on the wall next to the door and shown in Figure 13(a). The smaller circular valve controls the flow rate to the FT-IR. The N<sub>2</sub> flow rate is shown on the flow meter between the computer and optical bench (Fig. 13(b)). This flow meter is *not* good at measuring low flow rates and will be replaced at some point. The flow only needs to be enough to just get the ball moving a bit unless one needs a short-time fast purge.



**Figure 13.** (a) Main N<sub>2</sub> valve with blue swing handle. Round valve with label for FT-IR N<sub>2</sub> flow. (b) Flow meter.

**Detectors:** The standard IR detector is the room temperature DTGS (deuterated tri-glycine sulfate) detector. This is fine for 98% of typical samples run. We also have a high sensitivity liquid nitrogen (LN<sub>2</sub>) cooled MCT (mercury-cadmium-telluride) detector for use with the more IR-beam absorbing cells mentioned previously or special samples.

For highly demanding applications like the circle ATR solution cells one needs to make use of the high sensitivity broad-band MCT detector stored in Choppin 616. The MCT detector is ~10 times more sensitive than the DTGS detector! See Prof. Stanley for instructions on how to switch the DTGS and MCT detectors.

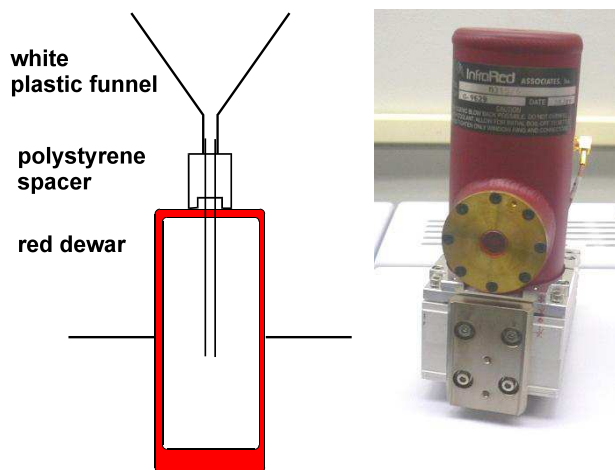
This detector has a small red dewar for the LN<sub>2</sub>. To use the MCT detector the red dewar needs to be filled with LN<sub>2</sub> using the plastic funnel with the 4" metal tube attached and the white polystyrene spacer shown in Figure 14.

Filling the MCT dewar takes a fair bit of time (~ 5-8 min) due to the time needed to cool down the funnel and dewar. Add the LN<sub>2</sub> slowly and minimize the spilling onto the FT-IR. Often right near the end of the cool down/filling process some of the LN<sub>2</sub> will “geyser” up out of the dewar, sometimes blowing the funnel out as well. This is normal and signifies that one is near the end of the dewar cooling stabilization process.

Usually before this geysering event the MCT detector has cooled down enough that the system reads it as functional. The FT-IR optical bench will “beep” and the STATUS light on the top right-hand side will change from red to green.

After filling the dewar remove the funnel and replace the small filler plug (which fits loosely) into the top of the FT-IR. The MCT detector is now ready to use. A filled dewar will last around 4-6 hours.

Each Tensor 27 FT-IR optical bench and computer is hooked up to an uninterruptable power supply (UPS) to protect it from power outages and surges.



**Figure 14.** Dewar schematic and photo of MCT detector.

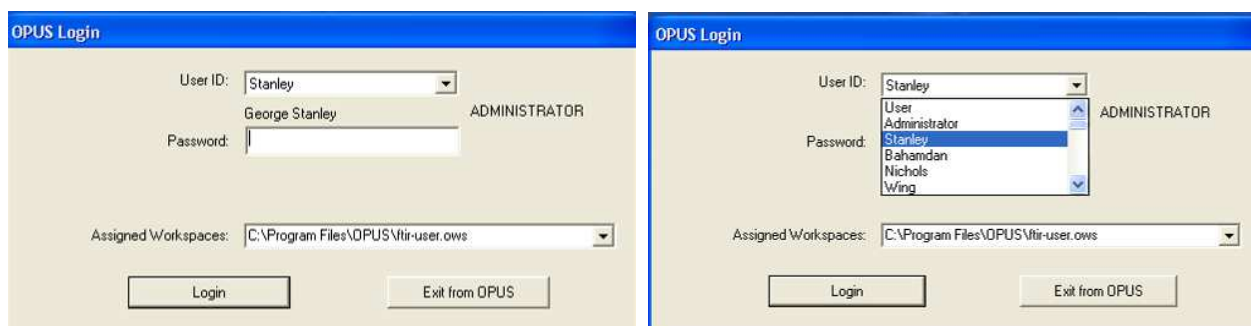
## Bruker OPUS Data Collection and Analysis FT-IR Software Program



OPUS (OPTical User Software) is the Bruker data collection and analysis program for the Tensor 27 FT-IR. Currently version 4.2 is installed on the computer in 702. Version 5.0 is installed on the computer in 616. Both are essentially identical in operation. We will be upgrading to version 6.5 in the near future. This section will then be updated as needed.

We have permission from Bruker to load OPUS on lab and office computers to view and analyze data away from the FT-IR. See Prof. Stanley for a CD. A full on-line manual is available via the HELP menu. Prof. Stanley has a hard copy in his office.

After starting OPUS the first thing you are presented with is the login screen shown below (left). The User ID's are listed in order of creation, not alphabetically. Find your name from the list (right screen), type in your password, and double check that the Assigned Workspace is as shown below. Then click on the Login button. It is possible to create your own workspace, but this is only for advanced users that are very familiar with the software.

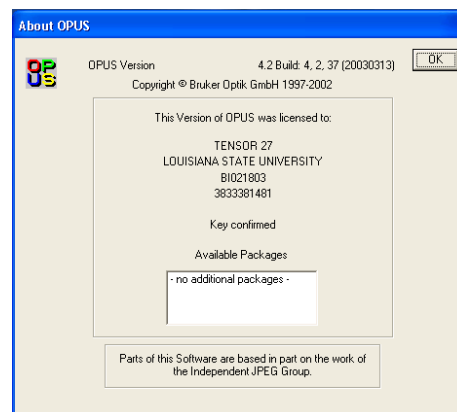


Bob Zinn or Greg O'Dell (Chemistry computer support personnel) need to setup an OPUS account for you. Make sure you let them know your faculty advisor's name so they can add that to the Windows user information. OPUS requires at least **Power User** privileges (or higher) in case you are setting it up on your own system. You should also have a user directory on the D: drive of the computer where you should store all your data.

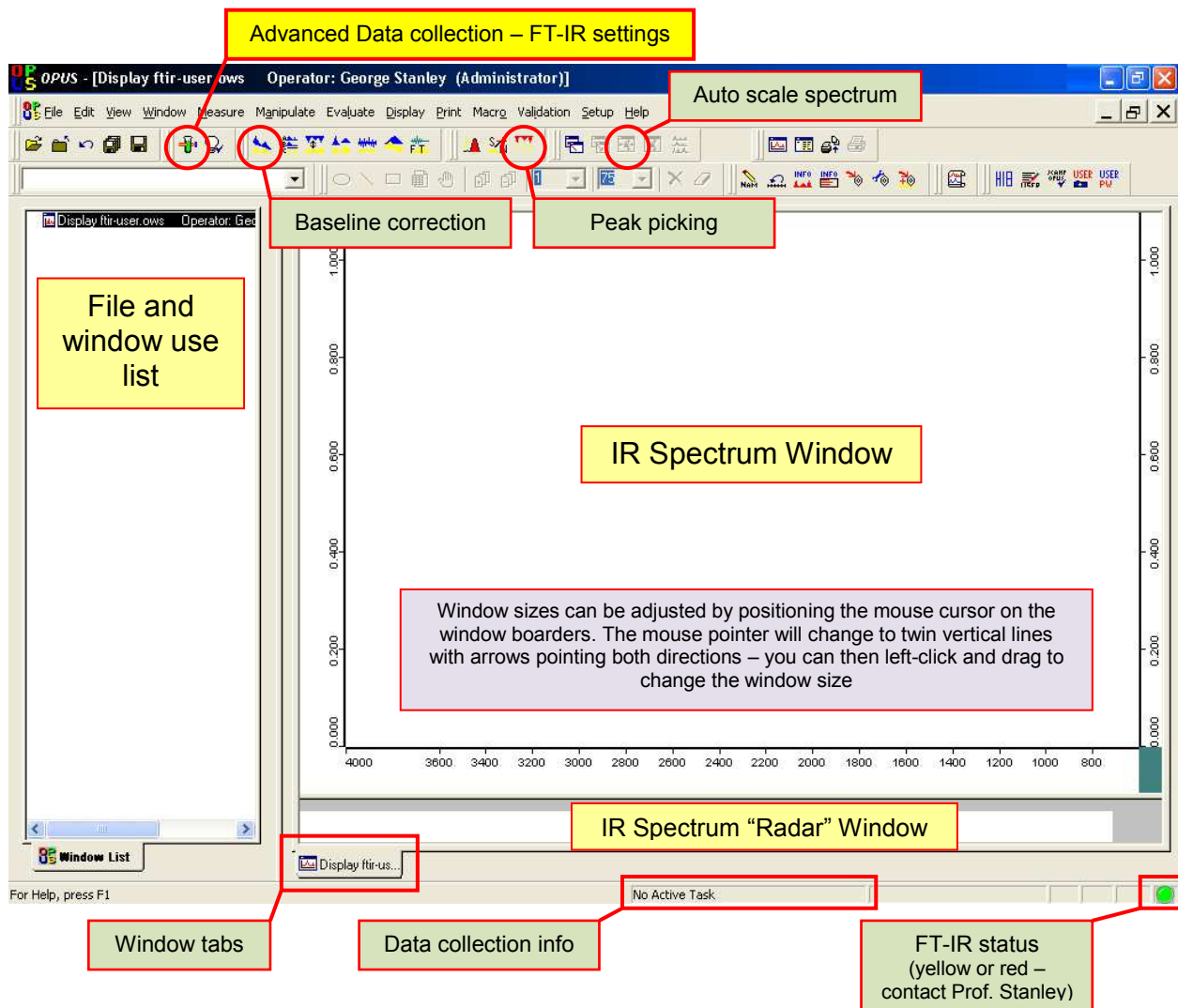
### Remember to always backup your data!!

If you forget your password (Windows or OPUS) please see Bob, Greg, or Prof. Stanley to reset it. Typing in the wrong password too many times for OPUS will lock the account.

After the Login screen there is a welcoming screen that listed information about OPUS (version #, license code, #, etc). Click the OK button to proceed.



The main OPUS window is shown below. Key icons and areas are labeled.



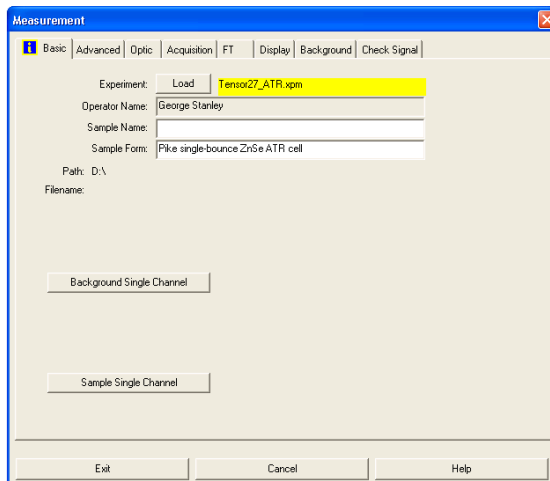
### Data Collection Details/Settings:



The data collection menus are opened by clicking on the icon shown or by selecting the **Measure** menu, then **Advanced Measurement**. This will open up the Measurement window with a number of tabs giving details on various aspects of the FT-IR, data collection, and FT processing.

The **Basic** tab lists the Experiment file being used to input the default FT-IR parameters for data collection. This file is called **Tensor27\_ATR.xpm**. The file is stored in **C:\Program Files\OPUS\XPM**. **If you see a different filename it is probably because the previous user loaded their own custom XPM file.**

When you setup a special experiment and change a number of parameters for data collection you can save the experiment as an XPM file to load again when needed. It is best to store this in your own data directory,



but some students store theirs in the program directory (try not to).

If you see some other experiment file loaded you should reload the **Tensor27\_ATR.xpm** file by clicking on the Load button next to Experiment and directing the program to the **Tensor27\_ATR.xpm** file in the **C:\Program Files\OPUS\XPM** directory (screen to the right). This XPM file is setup especially for the Pike ATR accessory.

If you are running salt plates (e.g., NaCl or KBr) pick that experimental file or just change the **Result Spectrum** (discussed below) under the Advanced tab to **absorbance** or **transmission** (not **ATR spectrum**).

The FT-IR does have Automatic Accessory Recognition (AAR) and I will experiment with this when we get the new OPUS v6.5 software installed and update these instructions. This feature recognizes the cell being used and loads the appropriate data collection parameters and background reference spectra. So you would directly add your sample and run the spectrum (no initial background needed). It is NOT active right now.


The **Sample Name** and **Sample Form** fields are text messages to identify your sample, phase (liquid, solid, film, etc.), and cell being used.

Next, click on the **Advanced** tab to bring up that screen. Although this screen is labeled “advanced” it has basic critical fields that you need to fill in. Importantly, this is the screen where you specify the **Filename:** that your spectrum and data will be stored into. Use an informative filename. OPUS will add a number as the file extension, starting with 0 (**your original data – do NOT delete**). It will auto-increment the extension # if you run a new spectrum and keep the same filename. Example:

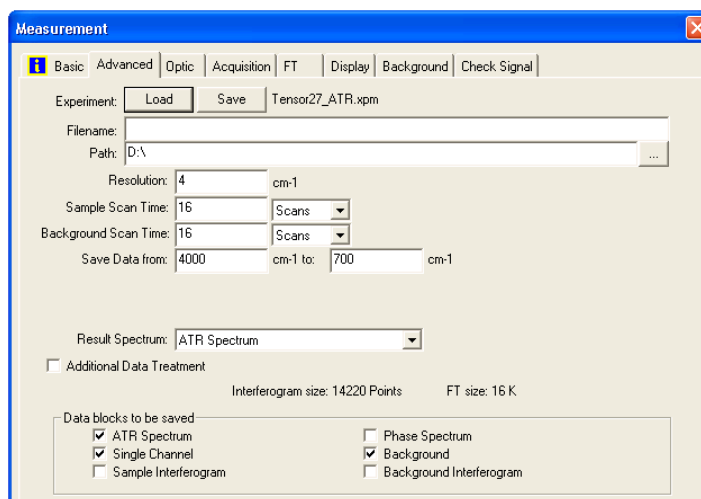
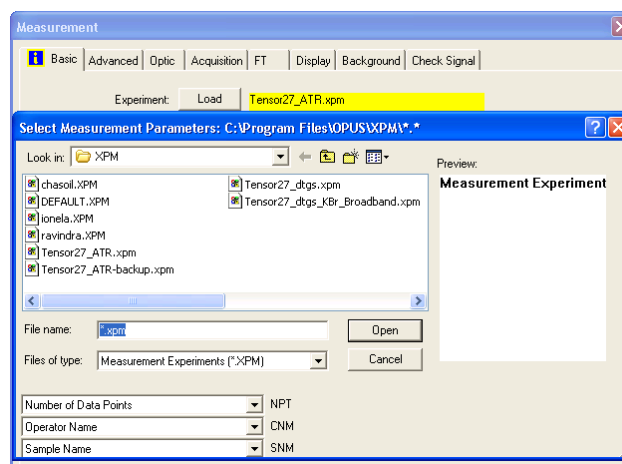
polystyrene.0  
polystyrene.1  
polystyrene.2

The **Path:** also needs to be checked and set to where you are storing your data!! This should be **D:\username** + any subdirectory name where you are storing your FT-IR data.

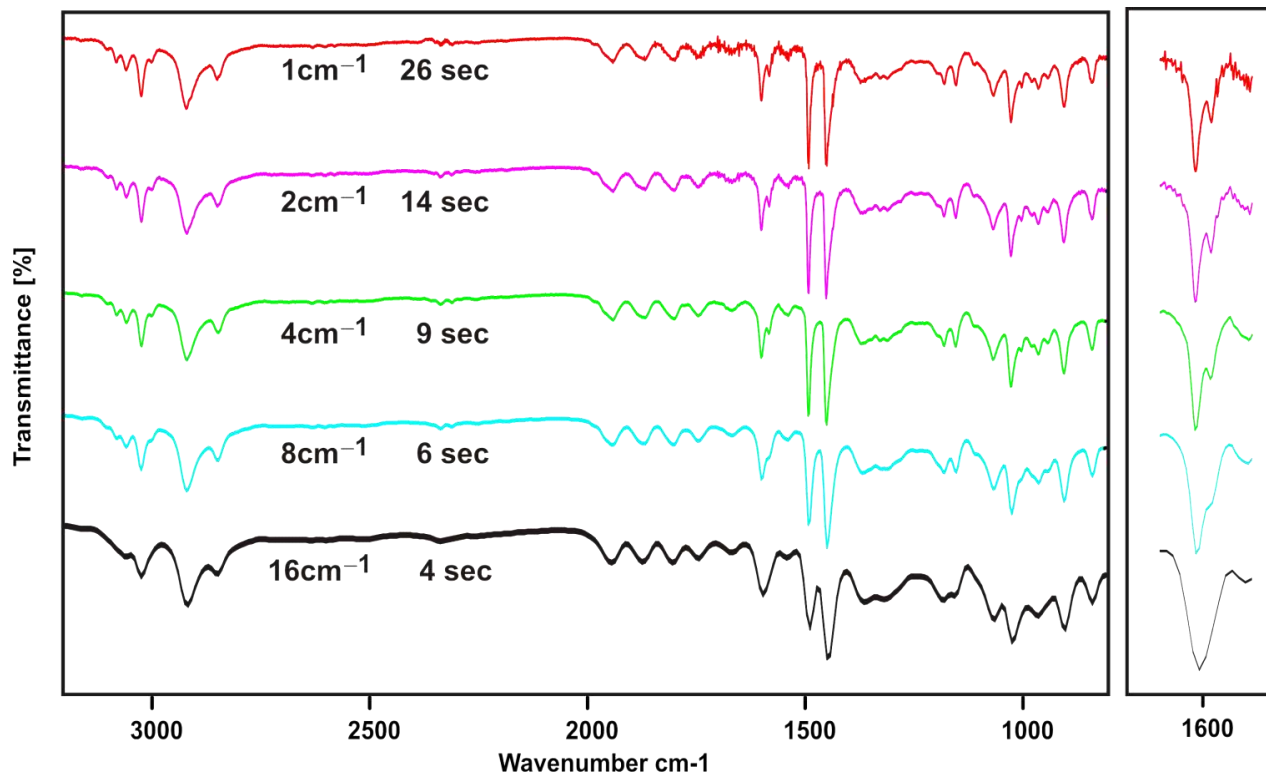
**Note that the user directory for the PREVIOUS user may be present!! So it is very important that you reset this path to YOUR data directory!!**

Clicking on the  button to the right of the **Path:** input line will bring up the file/directory selection window so you can direct the program to your data directory.

This “advanced” screen has almost all the common settings that you may want to adjust: resolution, # of background & sample scans, saved data range, type of spectrum (transmittance, absorbance), and data blocks that you want to save in the spectrum file. Descriptions of these key settings are discussed below.



**Resolution:** This is the instrument's spectral resolution in units of wavenumbers ( $\text{cm}^{-1}$ ). **The normal setting is  $4 \text{ cm}^{-1}$ .** Signal to noise in a spectrum is proportional to the square root of the acquisition time (# of scans). Doubling the resolution requires a four times higher acquisition time to achieve the same Signal-to-Noise ratio. Polystyrene film samples run on the Pike ATR cell at various instrument resolutions are shown below (with an enlargement of the  $1600 \text{ cm}^{-1}$  region shown to the right). These were not corrected for ATR (discussed later), but illustrate the resolution effect well. The times to collect **8 scans** at the **10 kHz scan speed** (standard for the DTGS detector) are listed with each spectrum.



The gross features of all 5 spectra are the same. Sharp peaks that lie close together, however, are adversely affected at the larger (lower) resolution settings. Note in the expanded view that the peak at  $1600.5 \text{ cm}^{-1}$  and its shoulder at  $1582.5 \text{ cm}^{-1}$  (which are  $18.0 \text{ cm}^{-1}$  apart) are well resolved in the 2 and  $4 \text{ cm}^{-1}$  resolution spectra, start losing definition in the  $8 \text{ cm}^{-1}$  spectra, and are pretty much smoothed together in the  $16 \text{ cm}^{-1}$  spectrum.

There are no differences between the 4 and  $1 \text{ cm}^{-1}$  spectra that justify the increased data collection time required at  $1 \text{ cm}^{-1}$  (or higher) resolution. Typically, one only uses the high resolution settings for gas phase spectra (we skipped the  $0.5 \text{ cm}^{-1}$  resolution option on this system since almost all our IR work is solution or solid phase). Solution and solid-state samples, for the most part, have broader peaks that certainly do not get any narrower by increasing the resolution setting on the FT-IR. If you do not have close lying sharp peaks and need faster collection times, the 8 or  $16 \text{ cm}^{-1}$  resolution options may be very good, but I would recommend running test spectra at  $4 \text{ cm}^{-1}$  resolution to make sure no important peak information is lost at the lower resolution settings.

The increased “noise” in the 1 and  $2 \text{ cm}^{-1}$  spectra arises in part from the non-perfect subtraction of the water vapor peaks from the background spectrum (the lower resolution settings broaden these sharp peaks leading to better subtraction), but mostly from the fact that we ran the same # of scans for each resolution. Remember that doubling the resolution requires a four times higher acquisition time to achieve the same Signal-to-Noise ratio.

**Background and Sample Scan Times:** We typically specify this with the # of scans (time in minutes is the other option). One almost always runs the same number of background scans as sample scans, so these should generally be the same. An unequal number of background and sample scans can lead to poor background subtraction (dramatically increased H<sub>2</sub>O and CO<sub>2</sub> atmospheric peaks, positive or negative going depending on conditions).

16 scans is the current default for the **Tensor27\_ATR.xpm** experiment file. For most “normal” concentration samples (pure liquids or solids) you will pretty much get an acceptable spectrum after only 1 scan. Since a scan at 4 cm<sup>-1</sup> resolution and 10 kHz scan speed only takes about a second, you can afford to collect 16 scans to get good signal to noise. Remember that signal to noise (S/N) in a spectrum is proportional to the square root of the acquisition time (# of scans). So if the S/N is not good enough, increase the # of scans by a power of 2 (e.g., 16 to 256).

For example, the effects of scan time (4 cm<sup>-1</sup> resolution, 10 kHz scan speed) for a dilute 10 mM solution of acetone in water are shown to the right using the Pike ATR cell (not good for dilute solutions!).

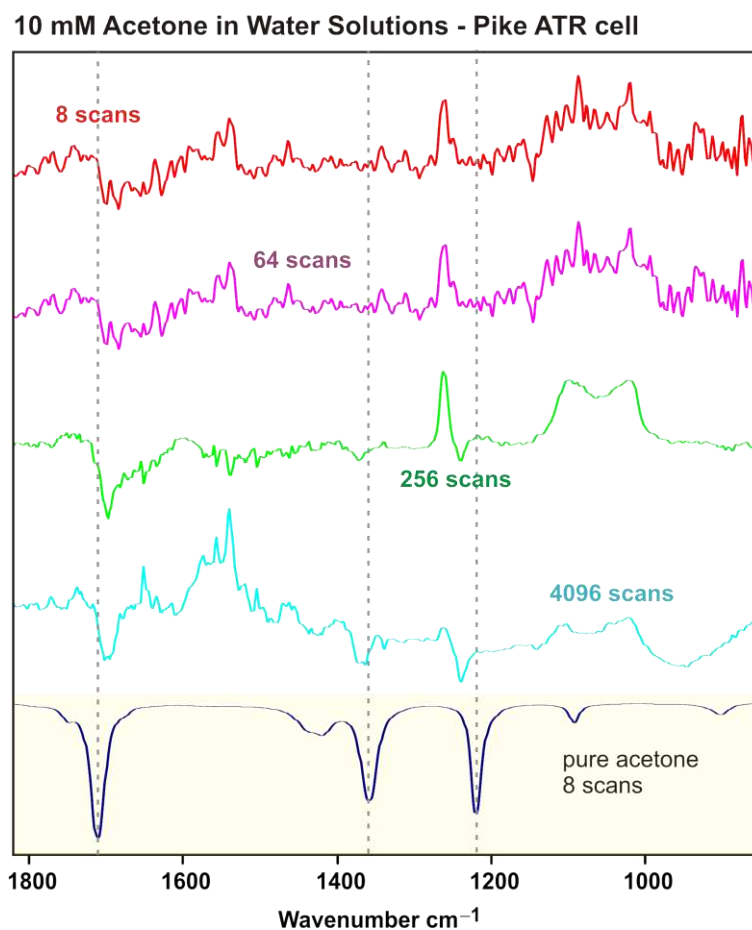
It is clear that the 8 and 64 scan spectra are not really showing anything. I would say that the 256 scan spectrum is starting to see the intense carbonyl stretch and maybe a few of the other strong bands. These bands are somewhat clearer in the 4,096 scan spectrum, but hardly what I would call good.

The spectra shown include an atmospheric H<sub>2</sub>O/CO<sub>2</sub> and aqueous correction (details later). This cleaned up the 256 and 4096 scan spectra a fair bit, but had little effect on the 8 and 64 scan IR's.

So having a concentrated enough sample is very important. Doing a lot of scans will not pull up a dilute sample from the noise or solvent subtraction artifacts unless you get lucky or have very strong bands.

**Save Data Range:** This represents the wavenumber range that will be saved and displayed for the spectrum. If you are using the Pike cell the ZnSe crystal absorbs most or all of the data below 650 cm<sup>-1</sup>. If you are using a KBr cell you can go down to 400 cm<sup>-1</sup>. The FT-IR is equipped with KBr beamsplitter so no data is available below 400 cm<sup>-1</sup>. There isn't much data above 4000 cm<sup>-1</sup> and this is the typically upper limit.

Note that the FT-IR interferometer physically scans from 8000 to 0 cm<sup>-1</sup>, but we only save a portion of that range.



**Result Spectrum:** This should be set on **ATR Spectrum** if you are using the Pike single-bounce ATR cell (or another ATR cell). The ATR mode records the IR spectrum in absorbance mode (peaks go “up” like an NMR spectrum) and applies an intensity correction to the spectrum. If you prefer transmission mode (peaks go “down”) you can easily change the ATR absorbance spectrum into transmission using the **AB <-> TR** conversion option under the **Manipulate** menu, or the



icon on the screen.

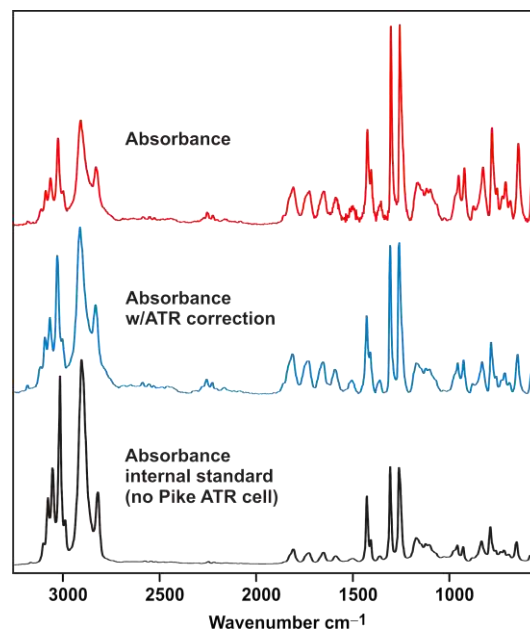
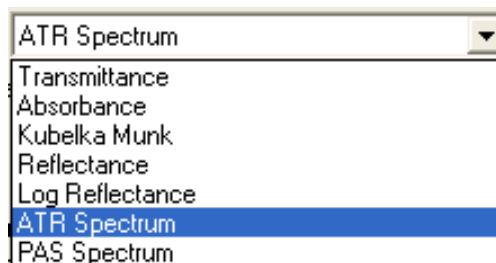
The penetration of IR radiation into the sample from the ATR crystal is wavelength dependent, with higher wavelengths getting less penetration and lower intensities. The ATR intensity correction partially compensates for this and is illustrated in the three polystyrene spectra shown to the right.

The top spectrum is a polystyrene film recorded on the Pike ATR cell in transmission mode, then converted to absorbance (8 scans,  $4\text{ cm}^{-1}$  resolution, 10 kHz). The second spectrum was collected the same way but using the ATR mode setting. The third spectrum is the internal polystyrene standard (no Pike ATR cell) collected in absorbance mode.

Note that the C-H region (around  $3000\text{ cm}^{-1}$ ) is considerably more intense than in the non-ATR corrected top spectrum. The ATR correction shown in the middle spectrum doesn't completely match the “correct” intensities shown in the bottom spectrum, but is a good compromise. Keep these intensity differences in mind when comparing your spectrum collected on the ATR cell to literature or database spectra that may be from a KBr sample (non-ATR).

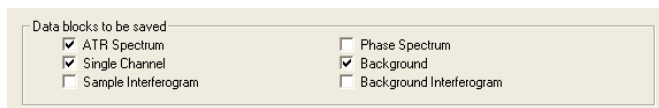
Other result spectrum settings include transmission (100% to 0%, peaks point “down”), absorbance, Kubelka Munk (corrections for diffuse reflectance spectra), reflectance, and photoacoustic (PAS) spectra. **If you have removed the Pike ATR cell to use the direct salt-plate/IR card cell then you should change Result Spectrum to absorbance or transmission.** Note that most of these corrections can be applied to a spectrum collected in one of the other modes (**Convert Spectra** option under **Manipulate** menu).

**Additional Data Treatment:** Generally not used at this point. I recommend doing any data treatments **after basic data collection** so you have a better idea of what impact you are having on the spectrum.





**Data Blocks to be Saved:** We typically save the spectrum, single channel (raw sample spectrum), and background. The spectrum (or trace) is the result of an automatic subtraction of the background spectrum from the single channel spectrum (sample + background). I strongly recommend saving both the single channel and background as they are both needed for any subsequent atmospheric ( $\text{H}_2\text{O}/\text{CO}_2$ ) data correction. If you want to be careful save the sample and background interferograms. These represent the raw data. All this information is saved in the file and the program can extract what you specify (more under data analysis & atmospheric correction).

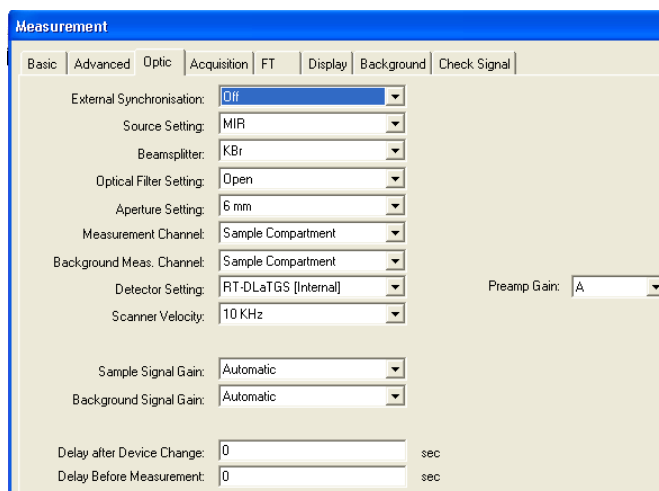


Data blocks to be saved	
<input checked="" type="checkbox"/> ATR Spectrum	<input type="checkbox"/> Phase Spectrum
<input checked="" type="checkbox"/> Single Channel	<input checked="" type="checkbox"/> Background
<input type="checkbox"/> Sample Interferogram	<input type="checkbox"/> Background Interferogram

**Optic:** Normally you don't have to change anything here, but you should always check it to make sure some previous user didn't screw things up by changing things and then saving the new settings over the **Tensor27\_ATR.xpm** experiment file.

I will only discuss a few of the settings that you might change under some circumstances.

**Aperture Setting:** Also known as the Jacquinot stop (J-Stop). This is an iris or aperture that is placed in the path of the IR beam between the source and the interferometer. The default setting should be wide open – 6 mm. Note that on the Tensor system in 702 aperture values up to 12 mm are listed, but 6 mm is the true largest setting. Do not set it larger than 6 mm.



Measurement	
External Synchronisation:	Off
Source Setting:	MIR
Beamsplitter:	KBr
Optical Filter Setting:	Open
Aperture Setting:	6 mm
Measurement Channel:	Sample Compartment
Background Meas. Channel:	Sample Compartment
Detector Setting:	RT-DLaTGS [Internal]
Scanner Velocity:	10 KHz
Sample Signal Gain:	Automatic
Background Signal Gain:	Automatic
Delay after Device Change:	0 sec
Delay Before Measurement:	0 sec

The purpose of the aperture is to select out only the center-most part of the IR beam where the IR radiation is the most parallel, or least divergent, as it enters the interferometer. This improves the resolution of the spectrometer for very narrow peaks. Naturally, this is only important for gas phase samples that have very narrow, highly resolved peaks. If one increases the resolution of the instrument to  $1\text{ cm}^{-1}$ , for example, OPUS will flag the Optic tab with a blue information tag and highlight the 6 mm aperture setting with a yellow background. This is the programs way of informing you that this setting should be changed to a smaller value to match your higher resolution. As you make the aperture smaller you are cutting down the intensity of the IR beam, so you need to watch the intensity under the **Check Signal** tab. The FT tab will also get an information mark to adjust some of those settings as well.

One practical use for the aperture is to reduce the intensity of the IR beam when using the highly sensitive MCT detector. The maximum # of counts of IR radiation that the detector can handle before producing “distortions” in the collected spectrum  $\sim 26,000$  counts (given as a negative # under **Check Signal**). Above this the detector is starting to saturate out. While many samples that demand use of the MCT detector will also drop the beam intensity enough, you may run into some samples where the MCT detector energy reading too high. In these cases one can close down the aperture to internally cut down the IR beam intensity to an acceptable level (under 25,000 counts).

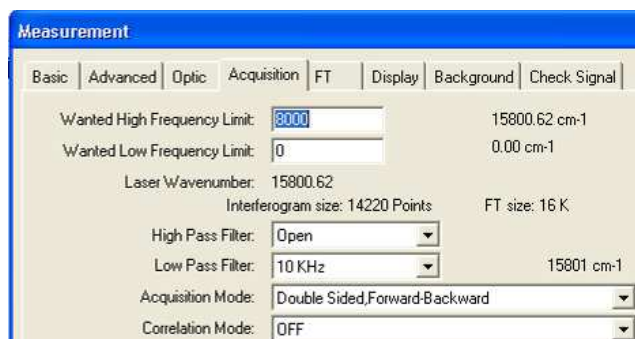
If you change the aperture, please remember to reset it back to the 6 mm settings for the next user! Unfortunately, some of the parameter settings get carried over to the next user if they are changed (despite loading in a fresh XPM parameter file).

**Detector Setting and Scanner Velocity:** The normal detector is the room temperature DTGS and the recommended interferometer scan velocity for this detector is 10 kHz. **Do NOT change these unless you use the MCT detector!!** If you need to use the liquid nitrogen cooled MCT broad-band detector the system will flag the detector setting to change. **For the MCT detector one should use the 20 kHz velocity setting.**

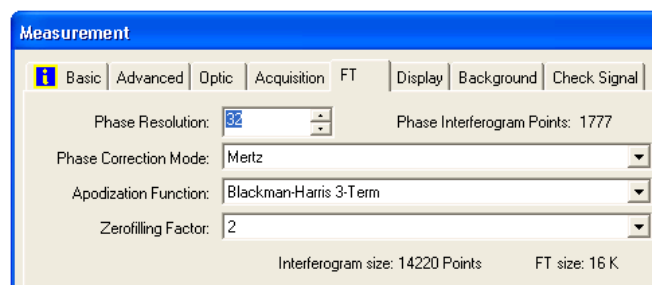
**Sample and Background Gain:** This sets the gain on the FT-IR internal electronics. Raising the gain increases the signal from the detector, but it also increases the background noise by a similar amount. There is little reason, therefore, to ever set the gain higher than 1 or automatic. If you need more signal to noise either collect more scans or use the MCT detector.

**Preamp Gain:** should be set to A. Do not change.

**Acquisition:** Normally you don't have to change anything here, but you should always check it to make sure some previous user didn't screw things up by changing things and then saving the new settings over the **Tensor27\_ATR.xpm** experiment file. All the settings should be as shown below.



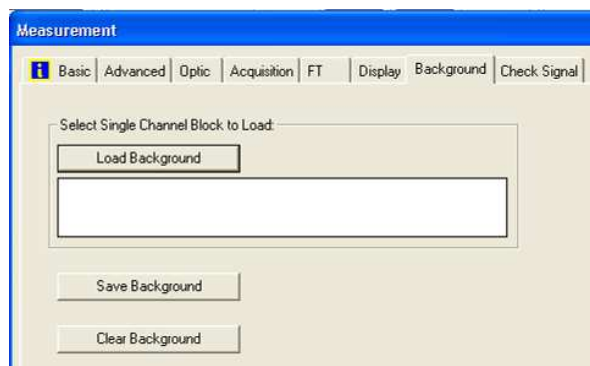
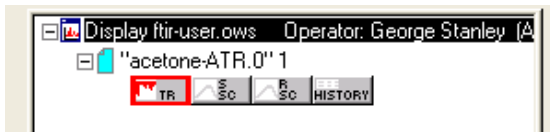
**FT (Fourier Transform) tab:** Normally you don't have to change anything here, but you should always check it to make sure some previous user didn't screw things up by changing things and then saving the new settings over the **Tensor27\_ATR.xpm** experiment file. All the settings should be as shown below. See on-line information or the manual for details on the settings.



**Display:** Normally you don't have to change anything here. The initial display will show wavenumbers from 4000 to 400  $\text{cm}^{-1}$ . This window typically tracks with what is set for the data collection save range (4000 to 700  $\text{cm}^{-1}$ ). The spectrum display window auto-adjusts to the spectrum range saved.

**Background:** Normally this is not used since you should usually collect a fresh background spectrum for each sample. BUT, if you have a saved background from a previous run that you want to load that can be done here.

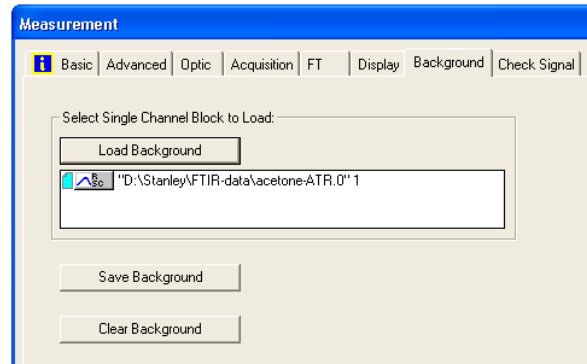
To load a background from a previous run you first need to open and load the spectrum file in question. Look carefully at the file and window pane on the left-hand side of the OPUS display. An example is shown below.



There are four boxes of data stored in the spectrum file. The first (highlighted in red) is the final sample spectrum or trace (TR). The next box represents the “raw” sample or single channel (S-SC) spectrum. The third box represents the reference or background single channel spectrum (R-SC). The last box is the History file that tracks anything you do to your original spectrum.

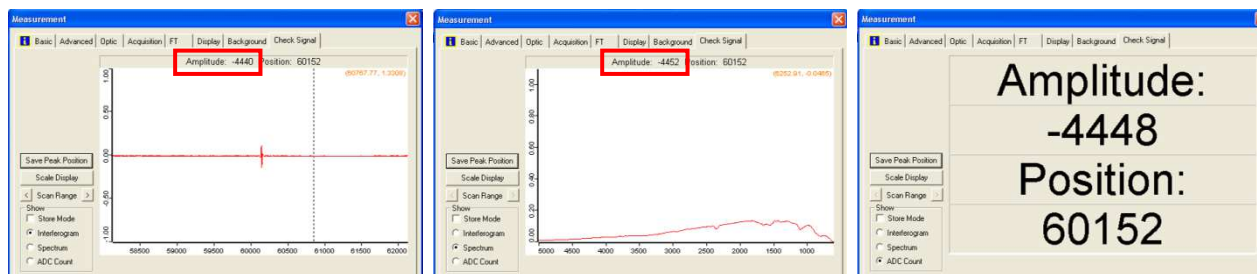
To load the background from this file you can take your mouse cursor and left click on the R-SC box and, while holding the mouse button down, drag it and drop it into the white box under the Load Background button. You will now see the display to your right.

Now you can click the “Load Background” button to put this background single channel spectrum into the FT-IR background memory slot. This background will then be used when you collect the next sample spectrum. If you actively collect another background it will replace this loaded background.



**Check Signal:** This is the other tab that you SHOULD check before every run!! This lets you know how much IR radiation the detector is receiving. Too low and you won't be able to record a spectrum or it will be filled with noise and no signal.

There are 3 types of displays available: interferogram, spectrum, and ADC count – all shown below.



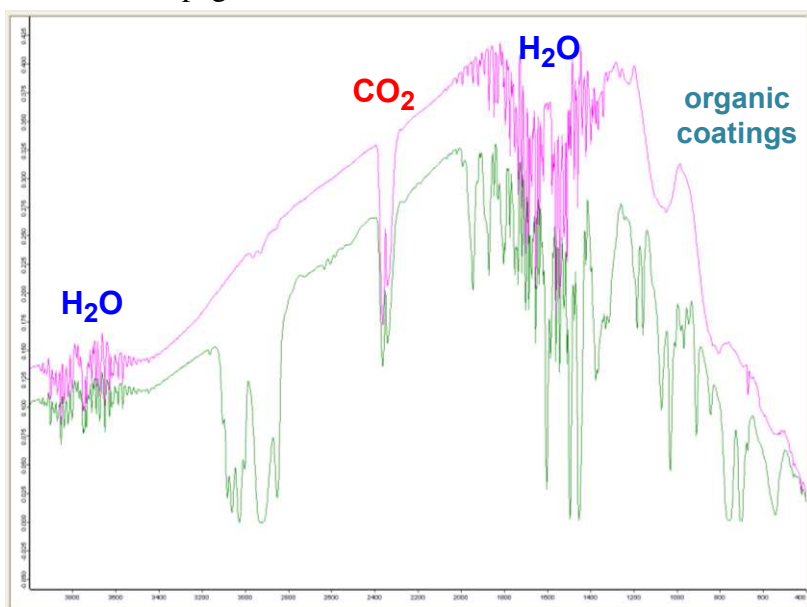
For the interferogram and spectrum screens the IR intensity is shown at the top of the screen as the **Amplitude**. The amplitude is listed as a negative number and the *absolute value* can vary from just above 3 to a high of about 18,400 counts (DTGS detector, standard settings).

Without the Pike ATR cell the IR intensity for the FT-IR system in Choppin 702 should be around 18,300 counts. With the Pike ATR cell in place and a good N<sub>2</sub> purged optical bench you should see an intensity of 4,400 to 5,050. If you see a significantly lower value, please contact Prof.

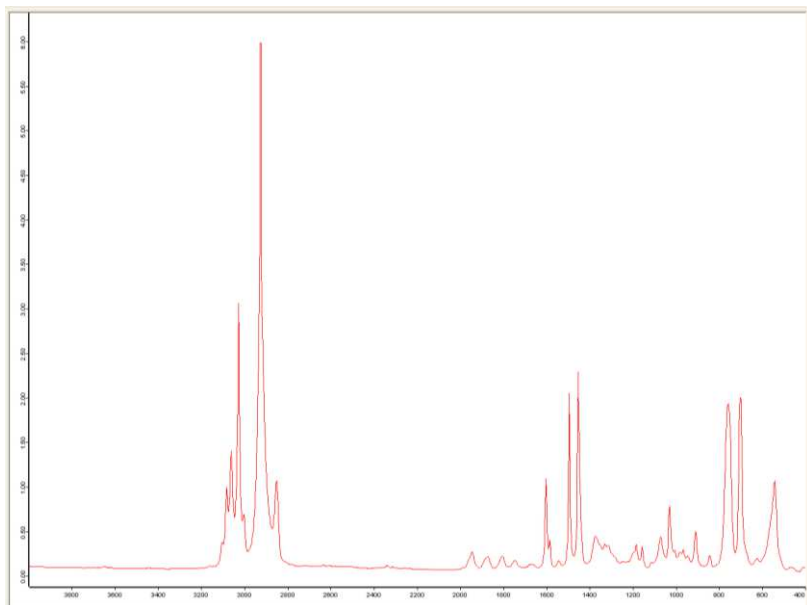
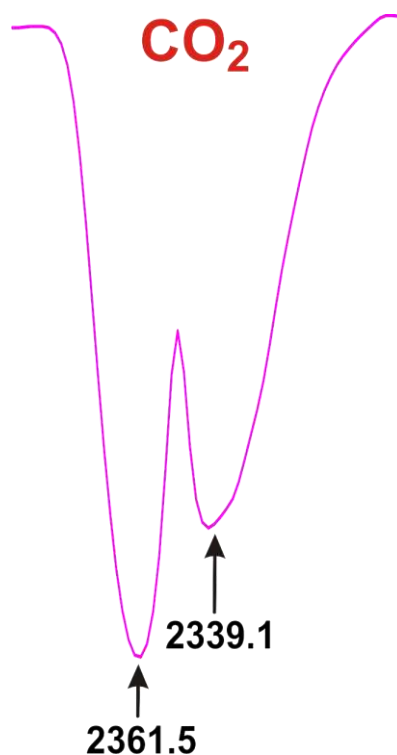
Stanley. Adding sample should drop the IR beam intensity as some is now being absorbed by the sample.

### Why Do You Have to Collect a Background Spectrum First?

Our FT-IR systems are single beam instruments. Air contains water vapor and CO<sub>2</sub> that are considerable absorbers of IR radiation. Water, in particular, has a host of sharp peaks in the gas phase and even in our air conditioned building the humidity level is still significant. The KBr window that isolated the source and interferometer compartments along with some of the other optics have organic coatings that also add to the background. Shown below are the single channel background (reference) spectrum (magenta) and “raw” single channel sample spectra (green) for polystyrene film. Note that the computer converts the interferogram to a transmission-type spectrum. The “sample” spectrum, as you can see, includes all the very significant background peaks, some of which I have labeled. The FT-IR internal computer subtracts these to generate the final sample spectrum (trace) shown at the bottom of the page.



The twin CO<sub>2</sub> peaks at 2361 and 2339 cm<sup>-1</sup> very commonly appear in your final sample spectrum. Do not mis-interpret these as part of your sample.



## Collecting a FT-IR Spectrum Using the Standard Pike ATR cell.

Here is an outline for what to do to collect an FT-IR using the standard Pike ATR cell.



- 11) Click on the Advanced Measurement icon:
- 12) **Basic Tab:** Check that the correct XPM experiment file is being used. This will usually be the **Tensor27\_ATR.xpm** experiment file. If someone else's experiment file is loaded, reload the **Tensor27\_ATR.xpm** experiment file (or your custom XPM file) by clicking on the Load button.

Measurement

Basic | Advanced | Optic | Acquisition | FT | Display | Background | Check Signal

Experiment: Load Tensor27\_ATR.xpm

Operator Name: George Stanley

Sample Name: isopropanol (wash grade)

Sample Form: Pike single-bounce ZnSe ATR cell

Path: D:\Stanley\FTIR-data\  
Filename: isopropanol-test2

- 13) **Basic Tab:** Type in a description of your sample

Measurement

Basic | Advanced | Optic | Acquisition | FT | Display | Background | Check Signal

Experiment: Load Tensor27\_ATR.xpm

Operator Name: George Stanley

Sample Name: isopropanol (wash grade)

Sample Form: Pike single-bounce ZnSe ATR cell

Path: D:\Stanley\FTIR-data\  
Filename: isopropanol-test2

- 14) **Advanced Tab:** Type in the name for the file that your IR spectrum data will be stored in. Make sure the path to YOUR directory is set.

Measurement

Basic | Advanced | Optic | Acquisition | FT | Display | Background | Check Signal

Experiment: Load Save Tensor27\_ATR.xpm

Filename: isopropanol-test2

Path: D:\Stanley\FTIR-data\  
Resolution: 4 cm-1

Sample Scan Time: 16 Scans

Background Scan Time: 16 Scans

Save Data from: 4000 cm-1 to: 750 cm-1

Result Spectrum: ATR Spectrum

Additional Data Treatment

Interferogram size: 14220 Points FT size: 16 K


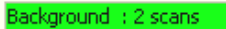
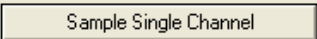

Data blocks to be saved:


ATR Spectrum  Phase Spectrum

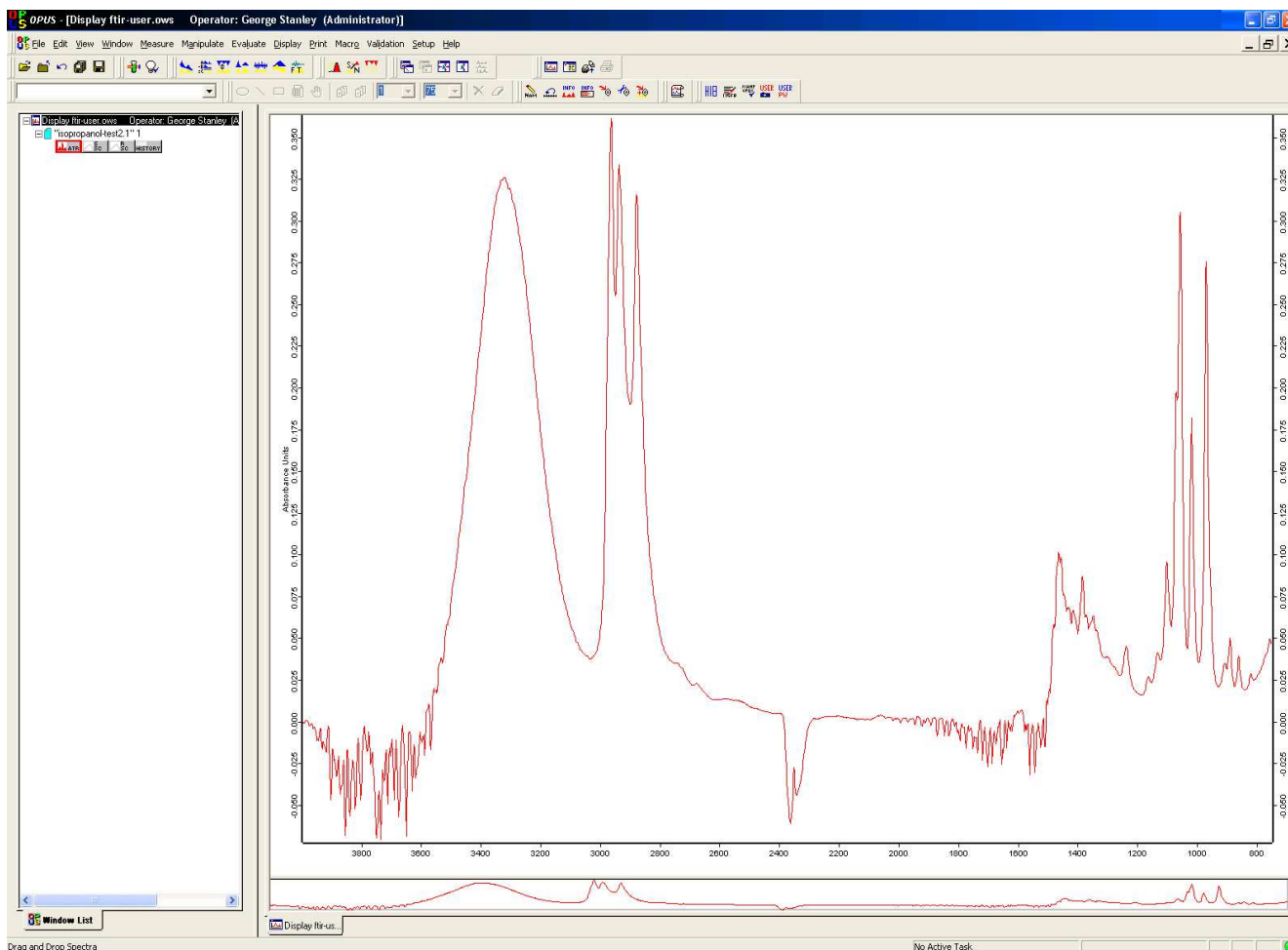
Single Channel  Background

Sample Interferogram  Background Interferogram

- 15) **Advanced Tab:** Check the resolution is set to 4 cm-1, background & sample scans = 16, result spectrum = ATR spectrum.
- 16) **Optic, Acquisition, and FT Tabs:** Check that these settings are OK (see previous section).
- 17) **Check Signal:** Check the IR intensity - should be around 4800-5000 counts.

- 18) **Basic Tab:** Run your background with the cell empty. After you click on the  button the FT-IR will start scanning the background. The progress of the scans is indicated in the bottom middle of the screen: 
- 19) **Basic Tab:** Add your sample to the crystal (see sampling details under Pike cell)
- 20) **Basic Tab:** Run your sample spectrum by clicking on the  button. The progress of the scans is indicated in the bottom middle of the screen: 

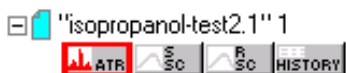
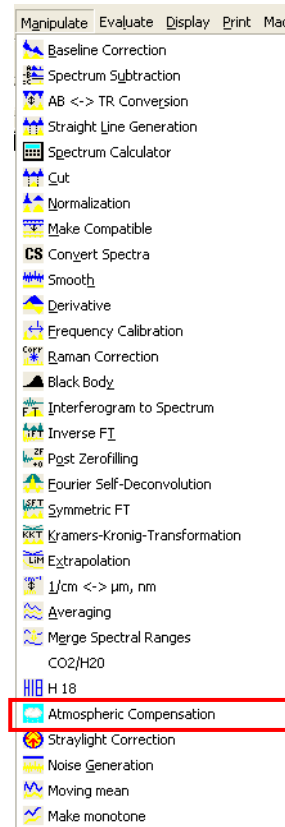
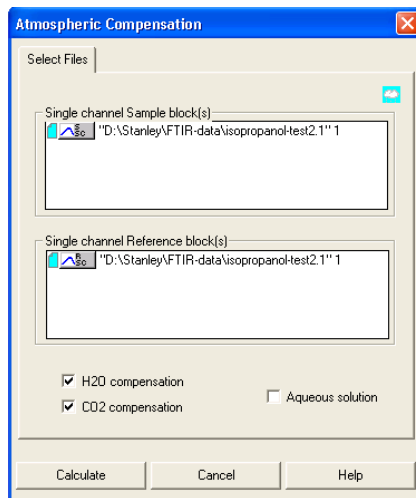
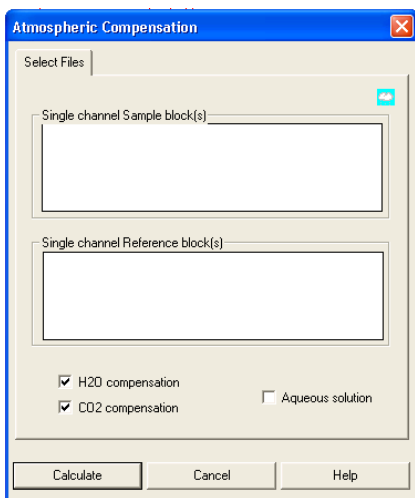
Your spectrum (ATM corrected absorbance mode) then displays (usually autoscales, but if not click on the autoscale icon:  )



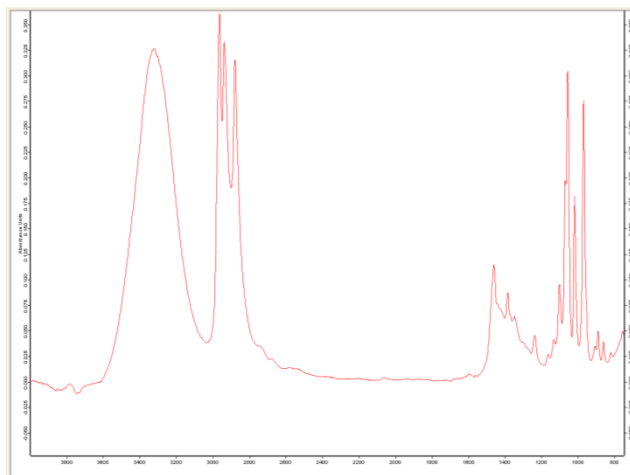
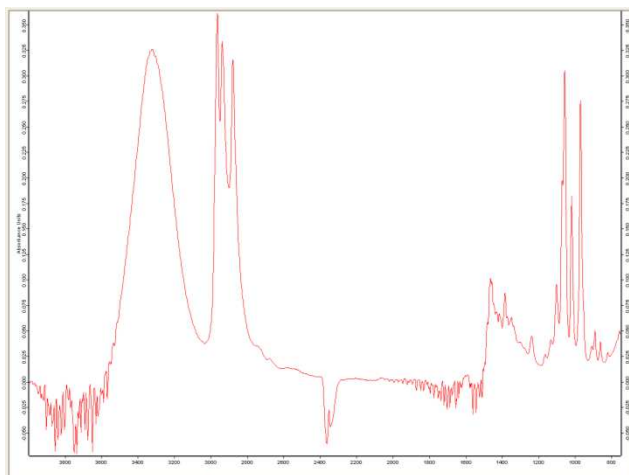
## Manipulating/Correcting your Spectrum

**Atmospheric Compensation (H<sub>2</sub>O/CO<sub>2</sub>):** Just by chance in the previous FT-IR there is a fair bit of negative going water and CO<sub>2</sub> absorbances present. This occurred because I was playing with the Pike ATR cell (removed and replaced several times) which let more water vapor and CO<sub>2</sub> into the cell itself. I then ran the background spectrum, BUT waited over 30 mins before running the sample. This allowed 30 mins of N<sub>2</sub> purging through the cell that reduced the amount of water vapor and CO<sub>2</sub> present when I finally ran the sample spectrum. Thus the original background spectrum that was subtracted from the sample + background spectrum had more water vapor and CO<sub>2</sub> present giving rise to negative intensity peaks (down-going) in the absorbance spectrum.

To do an Atmospheric Correction that should minimize the water and CO<sub>2</sub> bands, click on the **Manipulate** menu (shown to the right), then click on **Atmospheric Compensation**. That will bring up the empty screen (below left).




You now should left mouse click on and drag the sample single channel block (S-SC box) from the file display window and drop it into the top white box on the Atmospheric Compensation window. Do the same thing with the single channel reference block (R-SC) into the bottom section (screen, top right). Then click **“Calculate”**. Here is the spectrum before (left) and after (right) the correction:




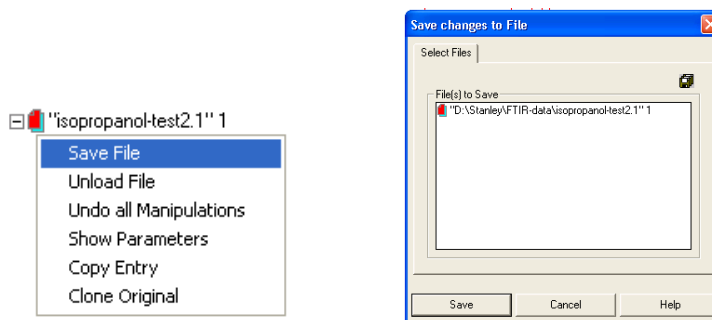
**There is also an option to apply a correction for aqueous solutions.**

Also note that after doing the atmospheric correction the file icon now shows a red page on top of the normal cyan color:



This indicates that the file has not been saved since the last spectral manipulation/correction. You can easily reverse what you did by clicking on the undo icon: 


The two quickest ways to save the spectrum file are right click on the red page indicator shown above, then select the Save File option (below left), or click on the file save icon, . Either of these brings up the final File Save option box (below right).



After saving the file, the red file marker goes away.

**IMPORTANT NOTE:** When you save a file after doing some sort of spectra correction OPUS creates a new version of the file with the modified spectrum by incrementing the numerical extension on the filename. So your original data is always stored in **filename.0**. The file with the highest numerical extension is the most recent version that contains all the data manipulations that you have done up to that point.


**Scaling/Zooming/Shifting/Peak Picking/Color:** Scaling and zooming the FT-IR spectrum is fairly straightforward.

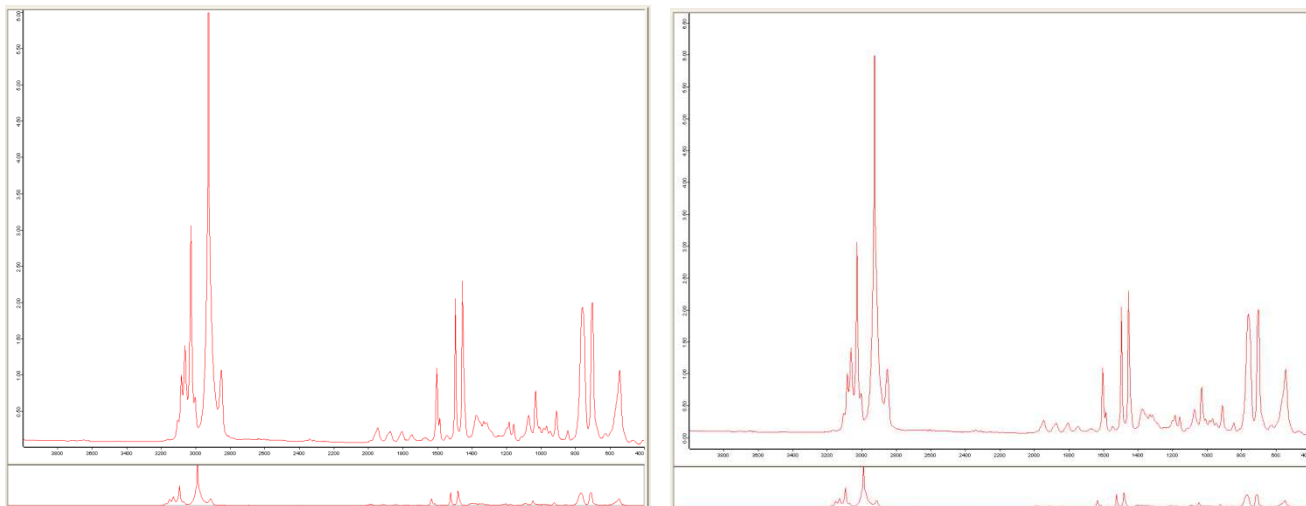
If the spectrum does not load with the maximum X and Y expansion, click on the autoscale icon  to expand the spectrum. **This is a very valuable button and is the quickest way to get you back to the full spectrum.**

The bottom window running across the main spectrum window shows a smaller, squished spectrum. This is the “radar” or overview window and can be used to adjust the main spectrum window. It is a bit tricky to use but good for fine adjustments. If you are working on a big enough display, I suggest doubling the size of this window vertically by clicking on the upper border and dragging it up to take away some room from the main spectrum window.

The “problem” with the autoscale button (function) is that it maximizes the vertical scale of the spectrum so the largest peak extends from bottom to top (absorbance type spectrum). This is typically too much if you want to do peak picking (next section) and list the picked peak frequencies on the top of the spectrum. The best way to add a bit more white space on the top or bottom of the spectrum is to use the overview window and increase the extent of the space around the spectrum.

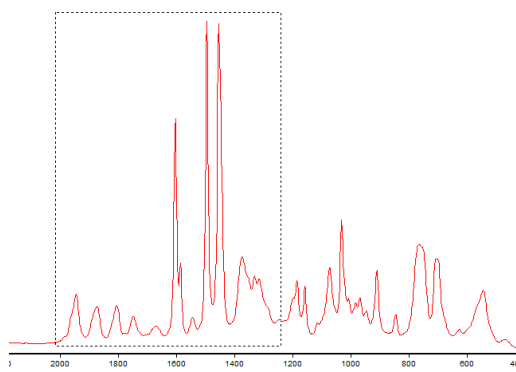
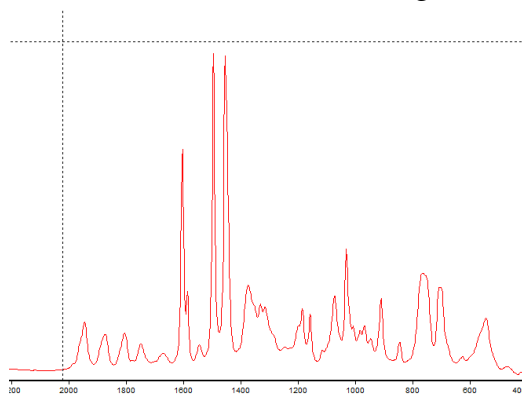
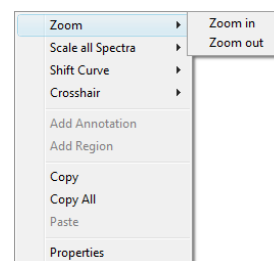


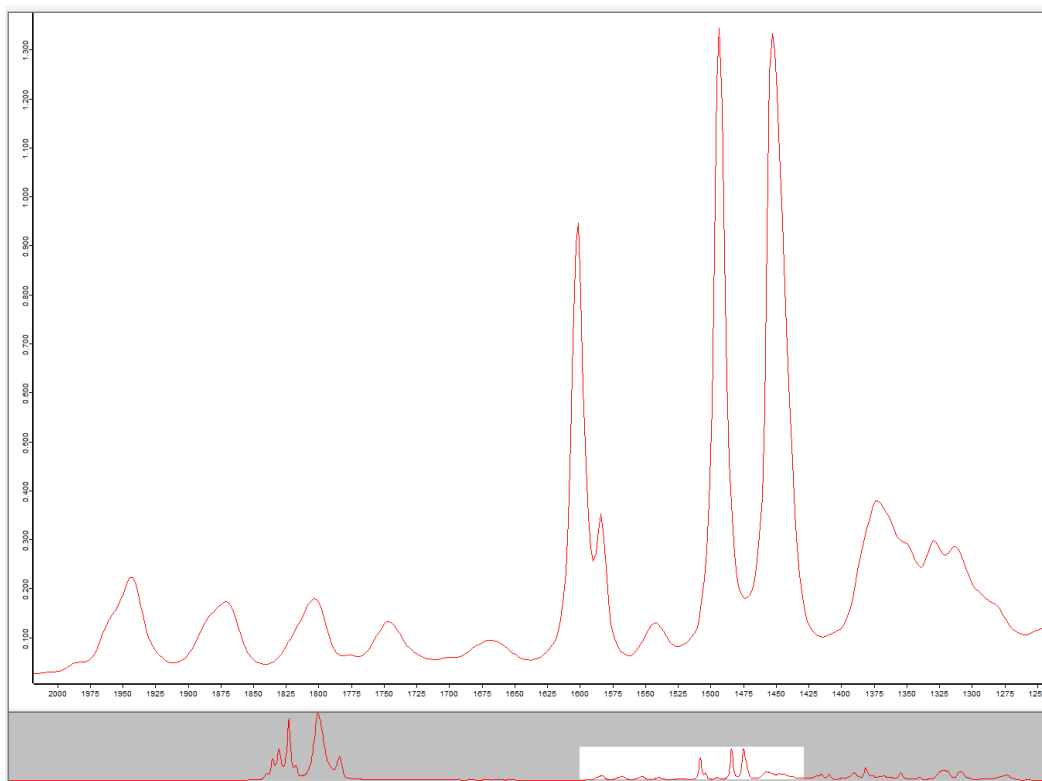
This is done by moving your mouse cursor to the top or bottom border of the overview window. When it changes to this pointer (  , or the single line version) you can left click your mouse and drag the white space around the spectrum to decrease it (make the spectrum in the main window more vertically expanded) or to increase it, which has the effect of reducing the vertical extent of the spectrum in the main window. **If you move the mouse pointer just a bit higher it will change to a horizontal double line with up and down single arrows. This is the cursor that allows you to change the size of the overview window itself.** Although the screen captures do not grab the mouse cursor the two views below show the effect of dragging the overview window white space up to make more room at the top of the main spectrum window. You can do the same thing on the bottom (which I did for the right side view) if you want to add a bit more white space there as well.




There is no button currently in the program that will increase or decrease the vertical scale in small increments. So practice with the overview window scaling features.


**Zooming** is pretty simple. Just right click on the main spectrum screen to bring up the menu shown to the right. Select zoom in and the menu will disappear and be replaced by vertical and horizontal dashed cursor tracking lines. Click to select the first defining point for your expansion region, then click the opposite corner point to define a square or rectangular expansion region. This does NOT immediately zoom the region selected, rather the dashed zoom box is now floating and will follow your cursor to allow you to position it more accurately around the part of the spectrum that you want to expand. Once you have it positioned correctly left click again to invoke the zoom. See images below showing the selection and the zoomed region on the next page.

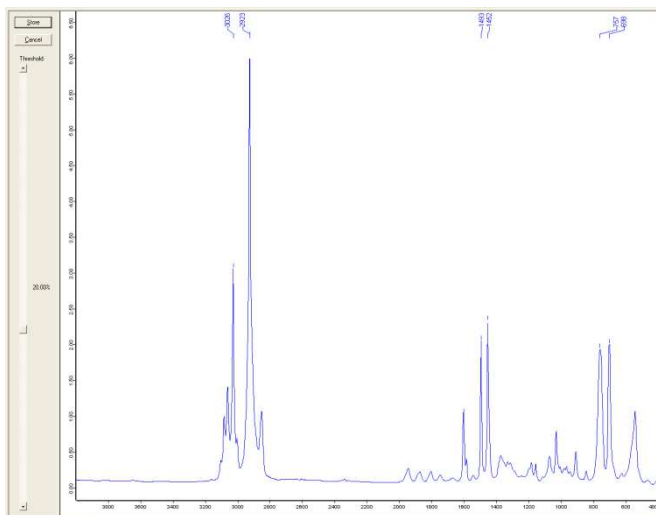
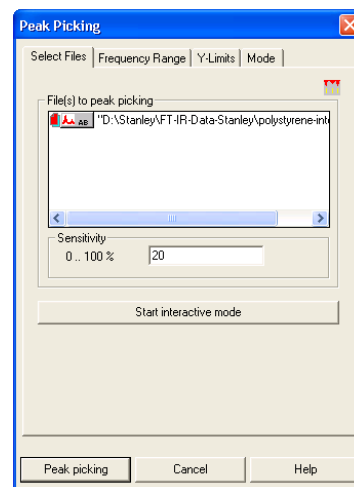




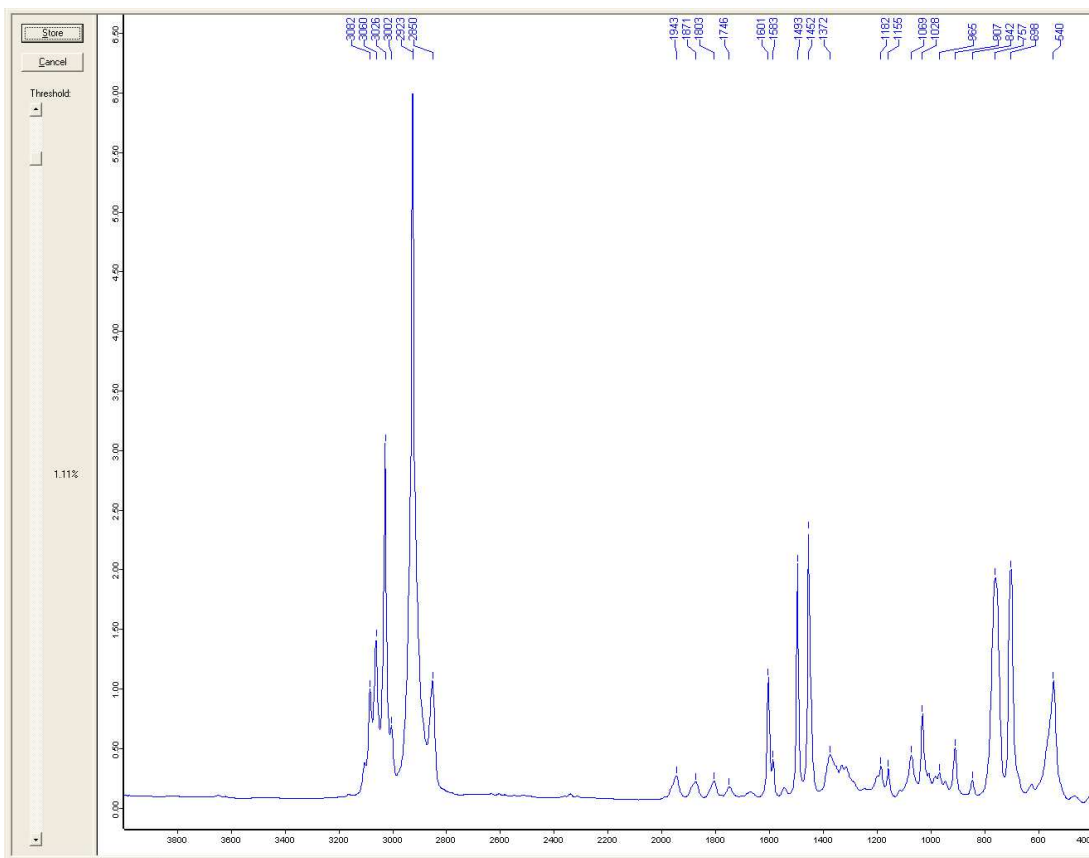
Notice the overview pane on the bottom of the screen. The zoomed region is shown with the white box. You can grab this white box with the mouse and move it around or resize the boundaries to change the zoom level!! By moving the box around you can scroll through the spectrum in a zoomed mode and fairly easily change the zoom level by changing the size of the white box in the overview window. That is why it is good to expand this window somewhat.

Once again, the quickest way back to the full spectrum is to click on the autoscale icon .

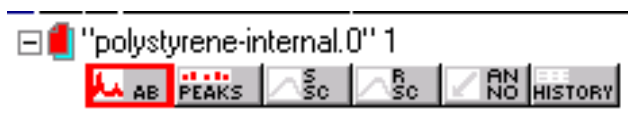
**Peak Picking** is also quite easy and is best done using the full spectrum. Click on the peak picking icon  or select **Peak Picking** from the **Evaluate** menu. This will bring up the peak picking screen shown to the right. **Interactive mode** is the best option, clicking on that modifies the main spectrum window as shown below.



By sliding the Threshold square up (more sensitive, more peaks picked) or down (less sensitive) you can determine what peaks are picked for labeling with the wavenumber. Note that the top numbers do NOT always appear on the display at this stage. What does always appear are the small vertical tick marks above peaks that are picked.



Once you have adjusted the threshold to get most of the peaks you want click the Store button and you will exit the peak picking mode and return to the main display with the peaks labeled with their wavenumber. At this point a new peaks data block is added to your spectrum file over in the file display window. This is shown below.



Right-clicking on this Peaks block and selecting the “Show Peak List” option will open a new display window with the peak pick list (shown below).

Display default.ows:1 Operator: Administrator

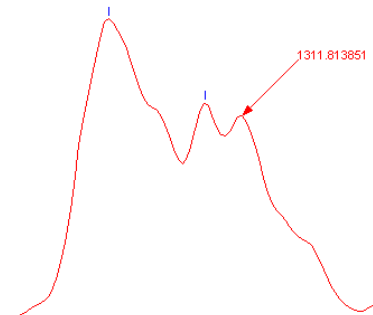
- polystyrene-internal.0' 1
- Report-Display - default.ows:2 Operator: Administrator
- polystyrene-internal.0' 1

"D:\Stanley\FT-IR-Data-Stanley\polystyrene-internal.0' 1

- Peak Table AB
  - Peak Picking

Wavenumber	Abs. intensity	Rel. intensity	Width	Found if threshold <	Shoulder
3081.9322	1.005	0.299	8.880	4.687348	0
3060.0071	1.427	0.790	10.692	12.807834	0
3026.1083	3.068	2.811	9.286	46.886314	0
3002.1234	0.700	0.170	8.034	1.303447	0
2922.9402	6.312	6.233	9.979	104.664177	0
2849.9648	1.076	0.554	16.315	9.114773	0
1942.8272	0.276	0.191	26.381	3.080393	0
1870.5221	0.224	0.129	30.399	2.130472	0
1600.5751	0.230	0.126	24.208	2.217012	0

If you want to mark a specific peak with a wavenumber label all you have to do is right click at the point where you want it annotated and select the “**Add Annotation**” option. This does not automatically pick the peak maximum, so it is best if you are zoomed in when you do this. This adds an arrow pointing at the point where you right clicked the spectrum and the wavenumber with a ridiculous number of digits after the decimal point. Right clicking on this annotation and selecting “Properties” allows you to edit the number and reduce the number of digits after the decimal point (one is sufficient). Left clicking on the annotation allows you to move it around.




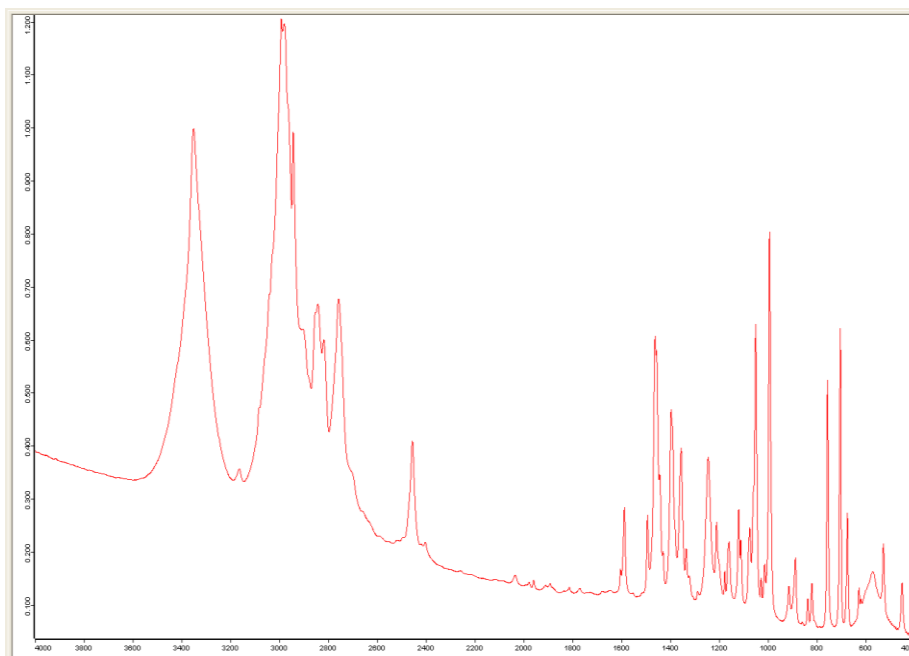
I haven't figured out how to increase the size of the font used as yet (but see later section on publication/talk/dissertation improved display options).

The **color** of the spectrum and peak picking marks/labels can be easily changed two different ways. The first is by left mouse clicking on the appropriate data block in the file window. Each time you click the color of that object will rotate around a pre-defined color pallet.

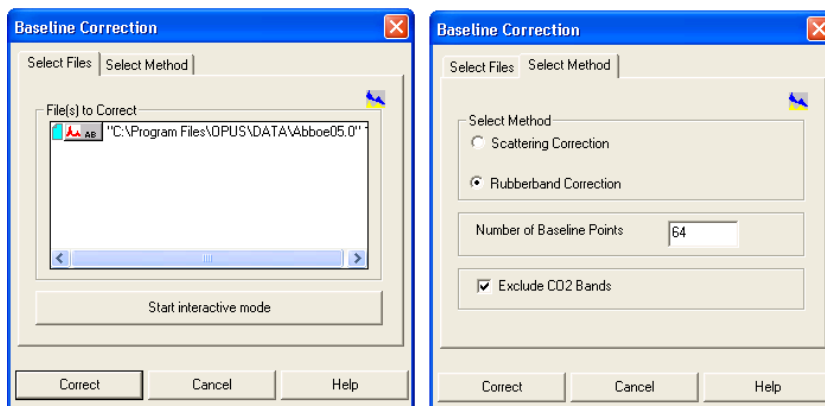
Want to more directly set a specific pre-defined color? Then right click directly on the spectrum and select the “**Set Color**” option. That will bring up the following screen with the colors available. Click on the color you want. This also works for the peak picking labels/tick marks. It does NOT work for annotations.



**Baseline Correction:** Samples that have sloped baselines can be “leveled out” using the **Baseline Correction** function under the **Manipulation** menu or via this icon . The following spectrum is a good candidate for baseline correction (automatic or manual).

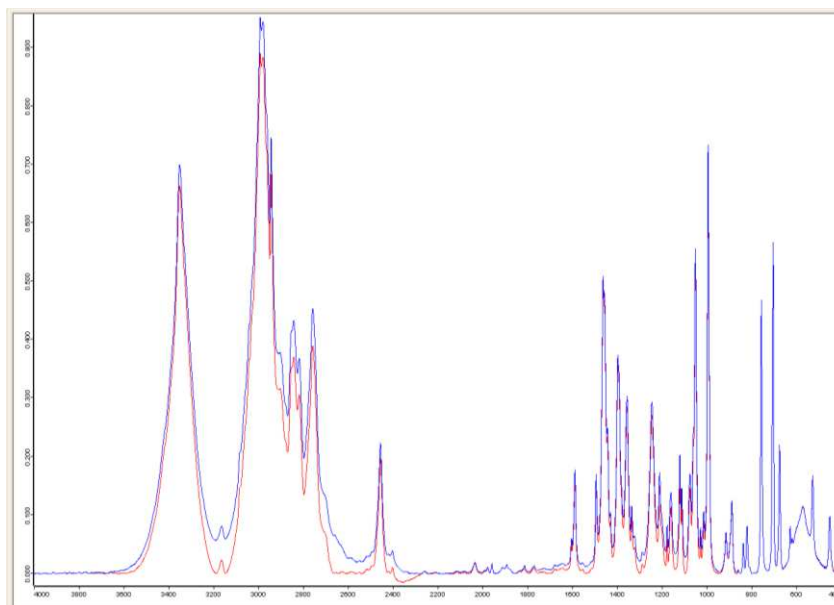


Invoking the Baseline Correction brings up the screen shown below (left). The Select Method tab gives the choices shown on the middle screen. The default **rubberband option** and 64 baseline points typically works well. Clicking on the Correct button starts the **automatic baseline** correction procedure and gives the spectrum shown below in blue.



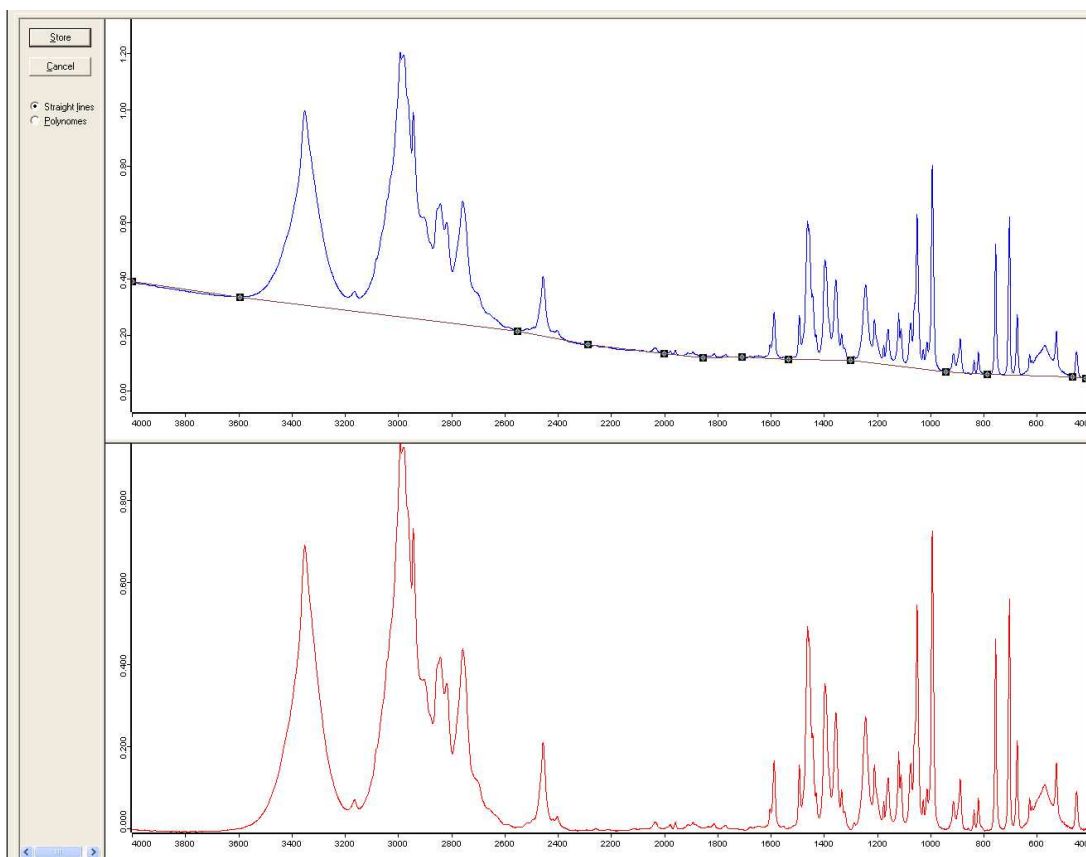
The **scattering correction** is more “aggressive” at pulling down the minima between peaks to the baseline. The automatic correction using the scattering option (64 points) is shown below in red.

To me the scattering option tries too hard at dragging peak minima down to the baseline. But I have to admit that it looks more like a classical IR spectrum. It is easy to try both. I would include which baseline option you use in your experimental procedure.




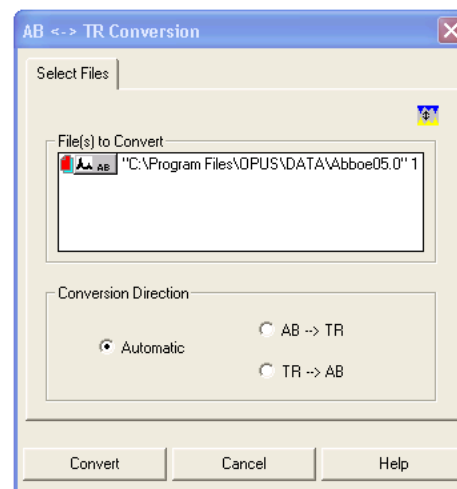
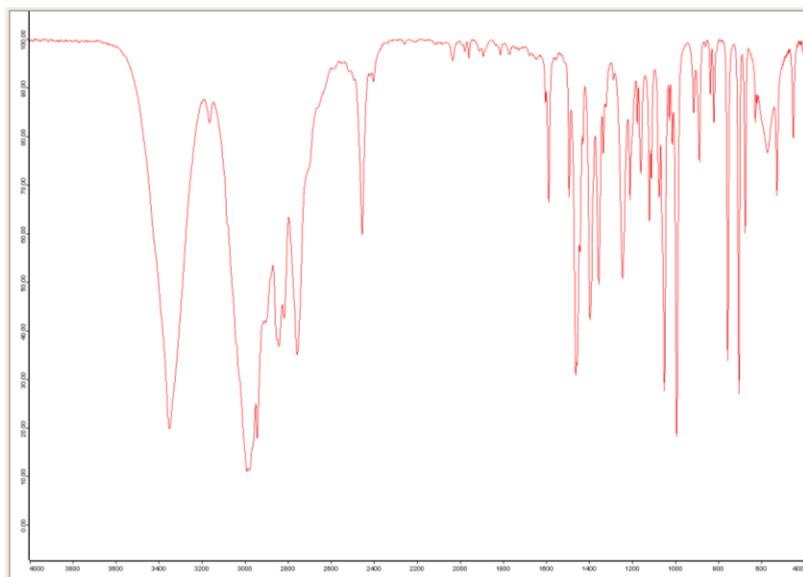
The **interactive option** should only be used when you want more control over the baseline correction or if the automatic correction doesn’t work well. **A key factor in doing the interactive baseline correction is to decide what part of the spectrum should be on the baseline!!** The “dips” between closely spaced peaks in the  $1400\text{ cm}^{-1}$  and lower region often do NOT touch the baseline due to overlap. Forcing them “down” via the interactive manual procedure when they should not be on the baseline could be considered “wrong” and improper data manipulation. So you need to be very careful when doing the manual interactive baseline procedure and not get carried away.

The interactive manual baseline routine changes the main spectrum window to give two spectra as shown below. The top window (blue spectrum in this case) is the one for double clicking on the baseline to defining the points that the spectrum will be pinned to for defining the baseline. A baseline point is defined when and where you left double mouse click. The points are NOT locked onto the spectrum and can be moved around once created. Double-clicking on an existing point will delete it. The figure below shows all the points I defined. The bottom spectrum represents the “corrected” spectrum based on the baseline points that you have added and is updated interactively as you add baseline points. Once you are happy, click on Store and you will be returned to the main spectrum window with the baseline corrected spectrum.

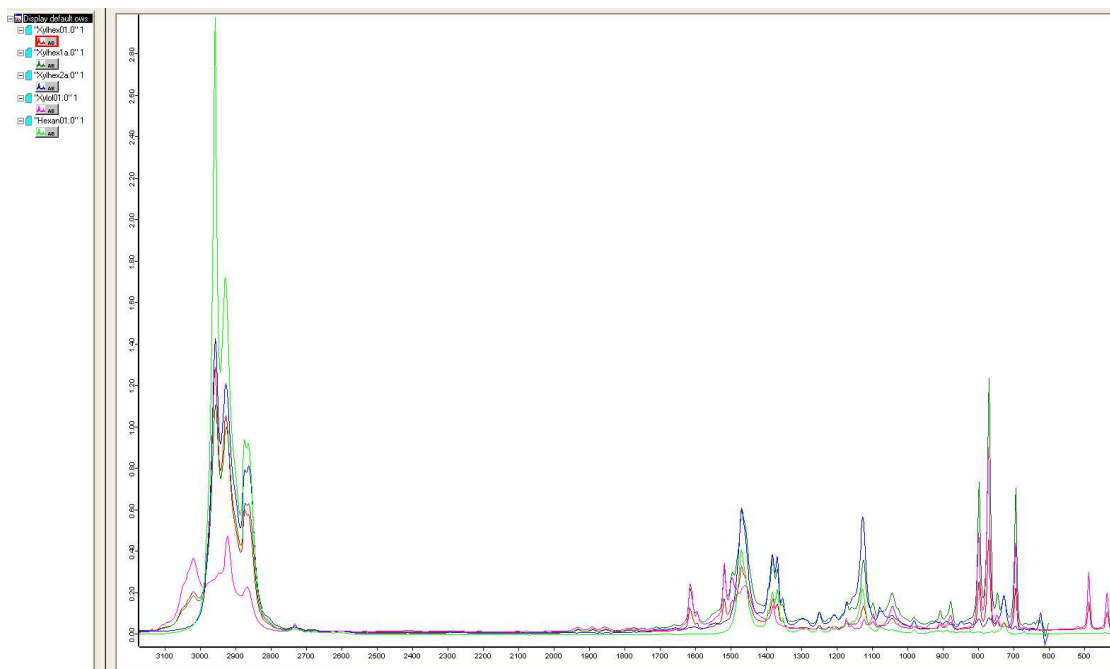



I typically use the [straight-line option](#), but the [polynomial correction](#) would be used for a baseline that has a lot of curvature in it. But you should experiment and see what works best for your spectrum.

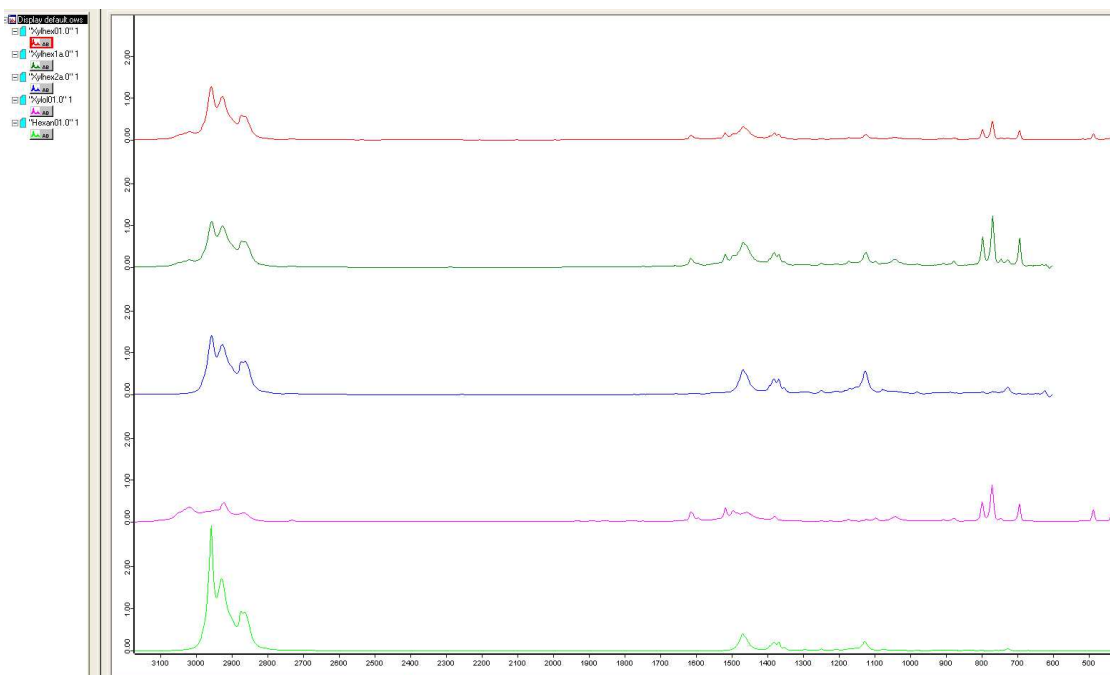
**Absorbance to Transmission Conversion:** If you like your spectrum presented as transmission (downward pointing IR peaks) then use the **AB <--> TR** function under **Manipulate** menu or via the  icon. The menu screen that opens up is shown to the right. The automatic mode generally works fine so all you have to do is click on convert. I converted the absorbance spectrum shown above (baseline corrected) to give the following transmission spectrum.



**Displaying Multiple Spectra:** The default mode when you load (or collect) more than one spectrum is **overlay** as shown below. I adjusted the spectrum colors to give reasonable contrast.




Clicking on the Display menu function **Stacked** or the **stack plot icon**  generates the perhaps more usable screen shown below. Clicking on the stacked icon again returns one to overlaid mode.

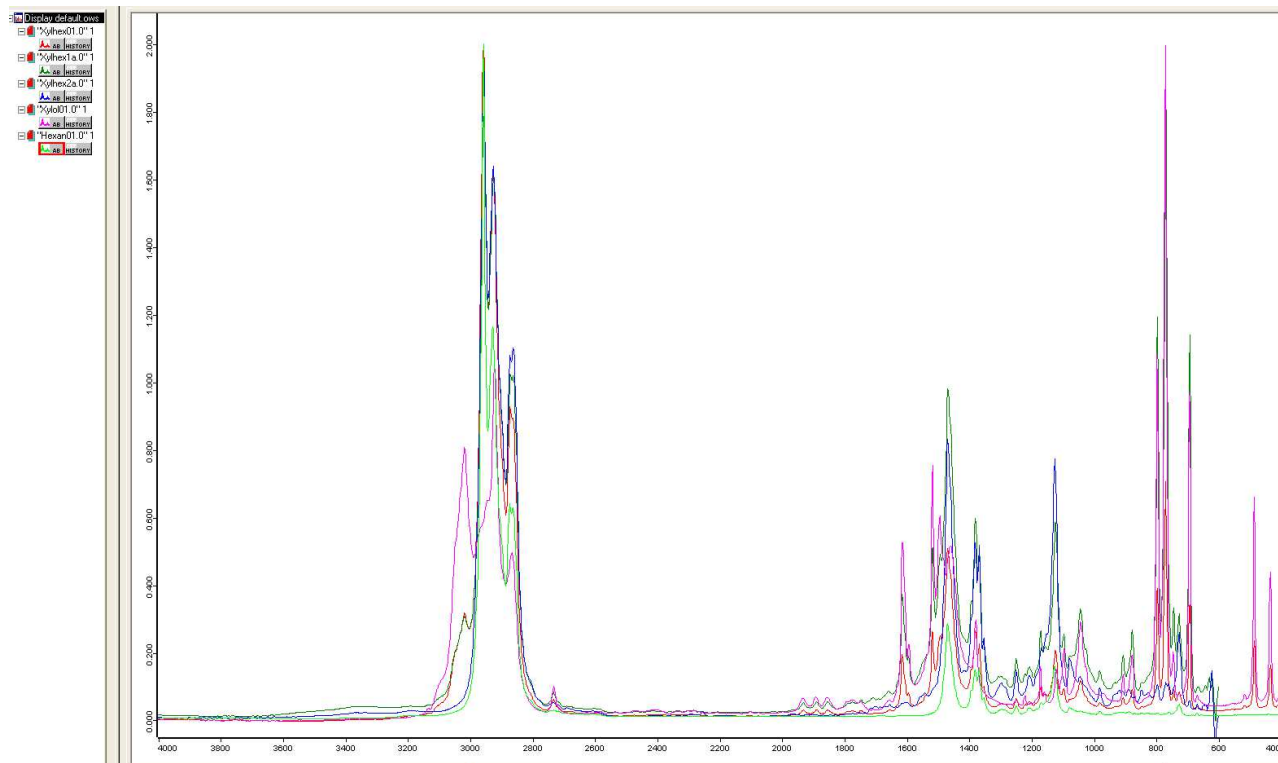
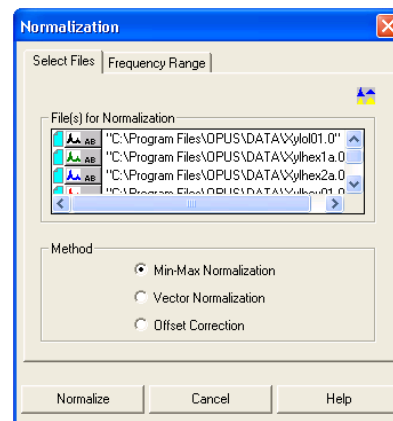


In overlay mode the display is scaled to the biggest peak in one of the spectra and there is no obvious display option to vertically expand one (or more) of the other spectra if they are less intense to see those peaks better. There are two ways around this.

The easiest method of interactively scaling overlaid spectra is to right click on the appropriate spectrum and select the “**Shift Curve**” option. There are three sub-options to shift curve: whole curve, top, and bottom. The top option allows you to click on one of the peaks and drag it (holding the left mouse button down) up or down. This, in effect, can individually scale the intensity of one of the overlaid (or stack plot) spectra. This is a “local” intensity change that is not saved with the spectrum. The trace can be easily returned to the original intensity using the “**Shift Curve – reset**” option. Note that the master XY rescale button will NOT sense any local intensity changes made using the Shift Curve options, that is it will behave as if you have not changed the intensity of the spectrum using the shift curve option. However, the Y rescale option does keep track of the shift curve intensity changes. You can also shift the bottom of the spectrum or shift the entire spectrum up or down with the other shift options.


The other method is to normalize all the spectra intensities using the **Normalize** function under the **Manipulate** menu or the normalize icon . That brings up the screen to the right.

By default this screen will only have **one** of the spectra listed, unless you have all the spectral files highlighted in the spectrum window. Otherwise, you need to click and drag the trace block of each of the other spectra over into the “Files for Normalization” window (which I have done). The Min-Max method generally works fine and what I used for this example. It normalizes each spectrum so the minimum absorbance is 0 and the max absorbance of the biggest peak in each is equal to 2. See the on-line help for more details on the other options. Normalizing all 5 spectra generated the overlaid display shown below.



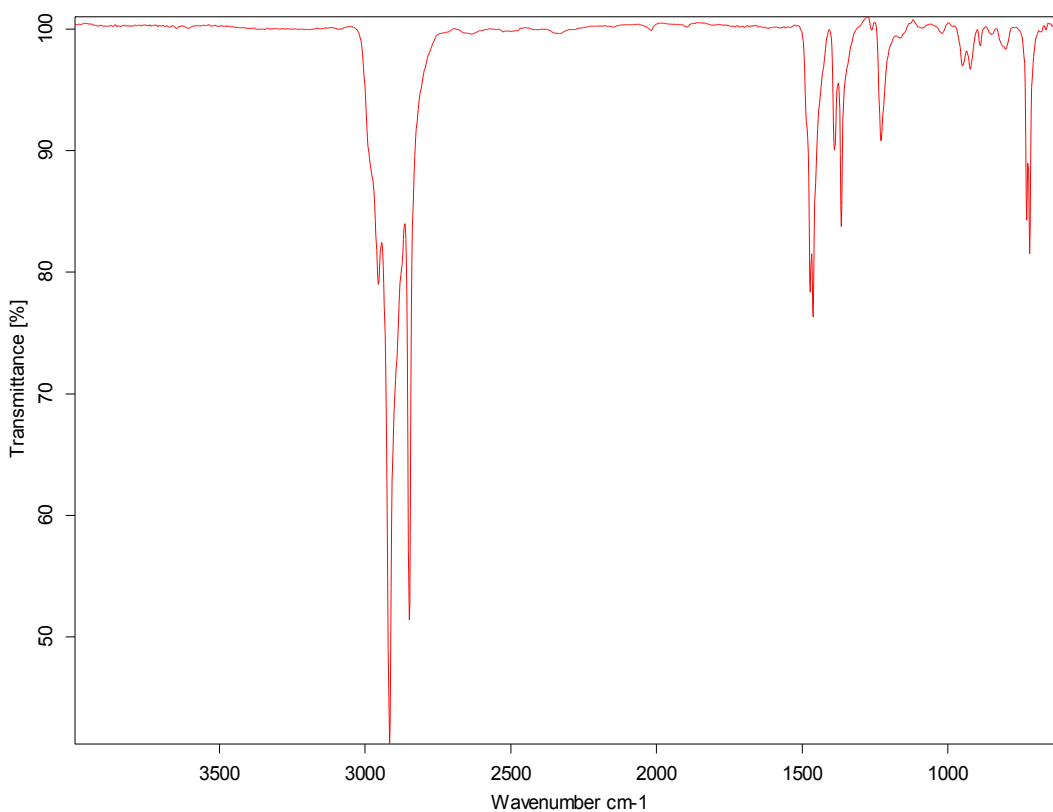
You should naturally NOT use normalized spectra for doing any quantitative calculations or comparisons between spectra. OPUS will save the normalized spectra with a new version #.



**Plotting Your Spectrum – Quick and Primitive (for your notes or advisor):** Just click on the Quick print icon  or select **Quick Print** from the **Print** menu. This works well for individual spectra (with or without peak picking) and overlaid spectra (with or without peak picking). **It does NOT currently work for stacked spectra (next version of OPUS may fix this).** I have extra ink cartridges for the inkjet printer in my office. Let me know when they need replacing.

A primitive (but effective) work around for getting a plot of your stacked plots is to press the Print Screen button on the computer (upper right side of keyboard). This does a bitmap screen capture and places it into the Clipboard. You can then open a new Word file and paste the image into it. Word has a cropping tool that you can use to trim the full screen capture to just what you want. This is how I got almost all the images into this document. Print the Word file and/or save it for future use.

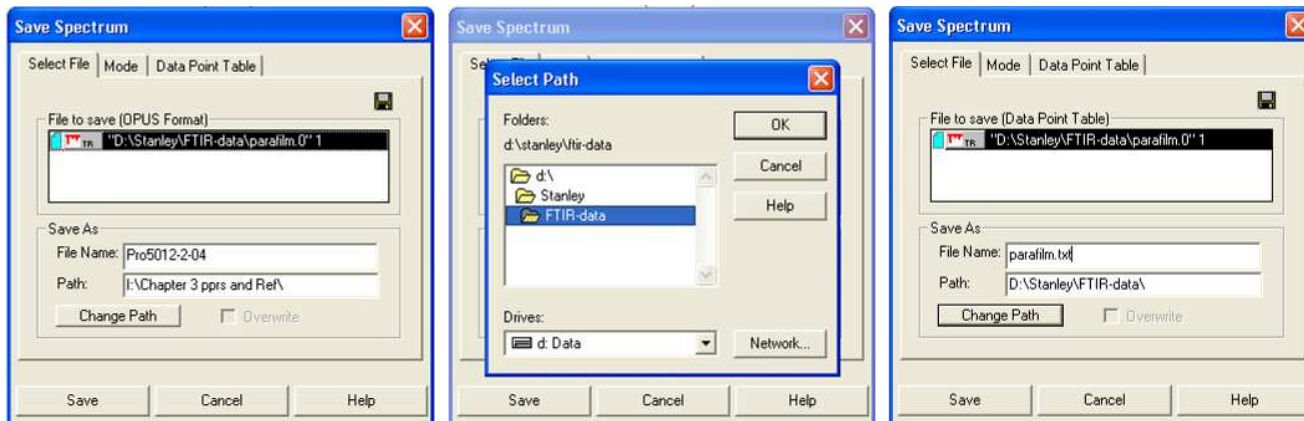
You can also **Copy** the spectrum to the Windows Clipboard and paste it into Word or most other applications. An example is shown below.



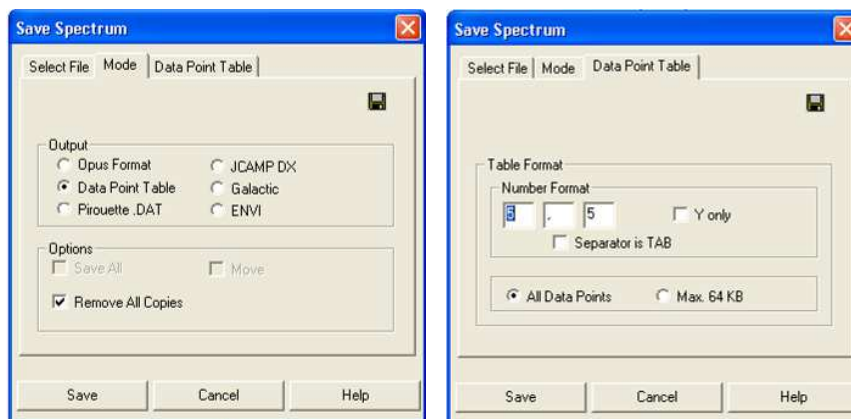
This is especially nice if you are keeping an electronic notebook. This is not a bitmap, but rather a vector graphics file and is quite compact. It can be edited using the Word drawing editing features and this will be discussed more later.

**Nice Spectra – Publication, Dissertation, Powerpoint Presentations:** There are several methods to take the basic plot(s) provided by OPUS and upgrade them to something suitable for publication (paper or dissertation) or presentation (e.g., Powerpoint). All of these involve some work and editing. **DO NOT SCAN IN YOUR SPECTRUM.** All methods below generate vector graphics files that have high resolution, small sizes, and don't become pixilated as you make them bigger.

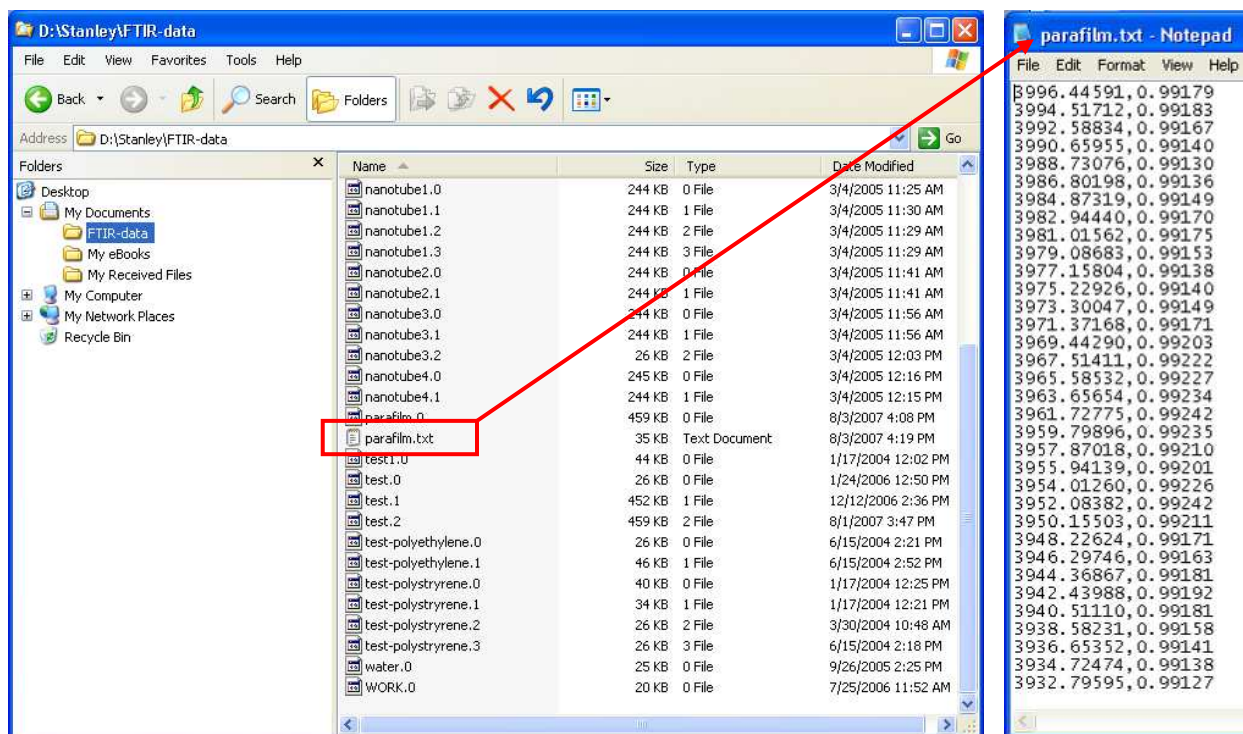
**Export Data as ASCII X,Y Data File – Replot with Plotting Program.** Exporting the data from OPUS as an X,Y data file is quite simple and shown below. With your spectrum on the screen click on **File** menu, then **Save File As**. This will bring up the first screen shown below left. Change the path to your directory (middle screen), then type in the file name and use a TXT extension. **You can only export ONE spectrum at a time.**



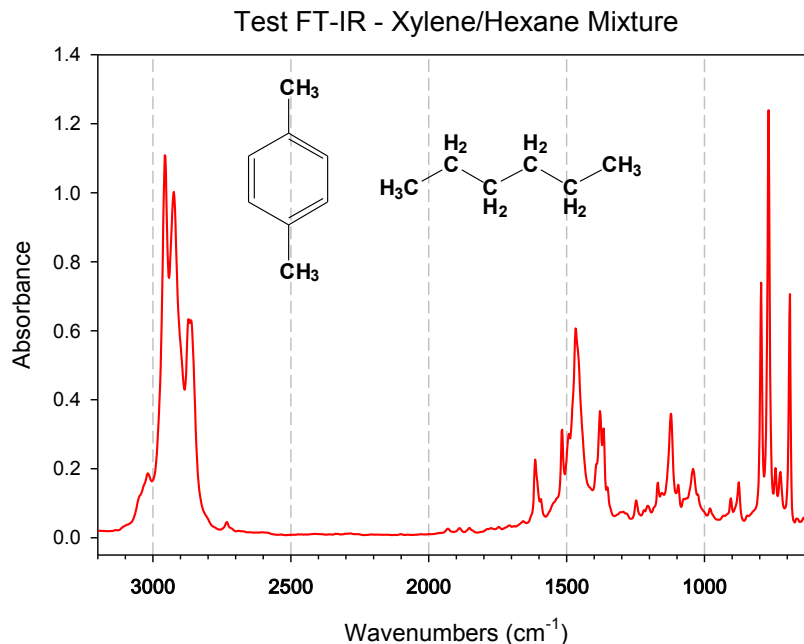
Then go to the **Mode** tab and click on **Data Point Table** as your file format. The Data Point Table tab shows the format for your data. The number format refers to the # listed after the decimal point. You don't need 5 decimal points for the wavenumber or intensity, but I usually leave it set at 5.



The screen below shows the text data file created (parafilm.txt) and that file opened with Notepad showing the X,Y data pairing as wavenumbers and intensity.



You can then import this X,Y text file into a plotting program such as Origin or SigmaPlot, re-plot the data and fine tune the graph to your satisfaction. I used the SigmaPlot (9.0) program to import some FT-IR data and a sample plot with ChemDrawings added is shown below (cut and pasted into Word).

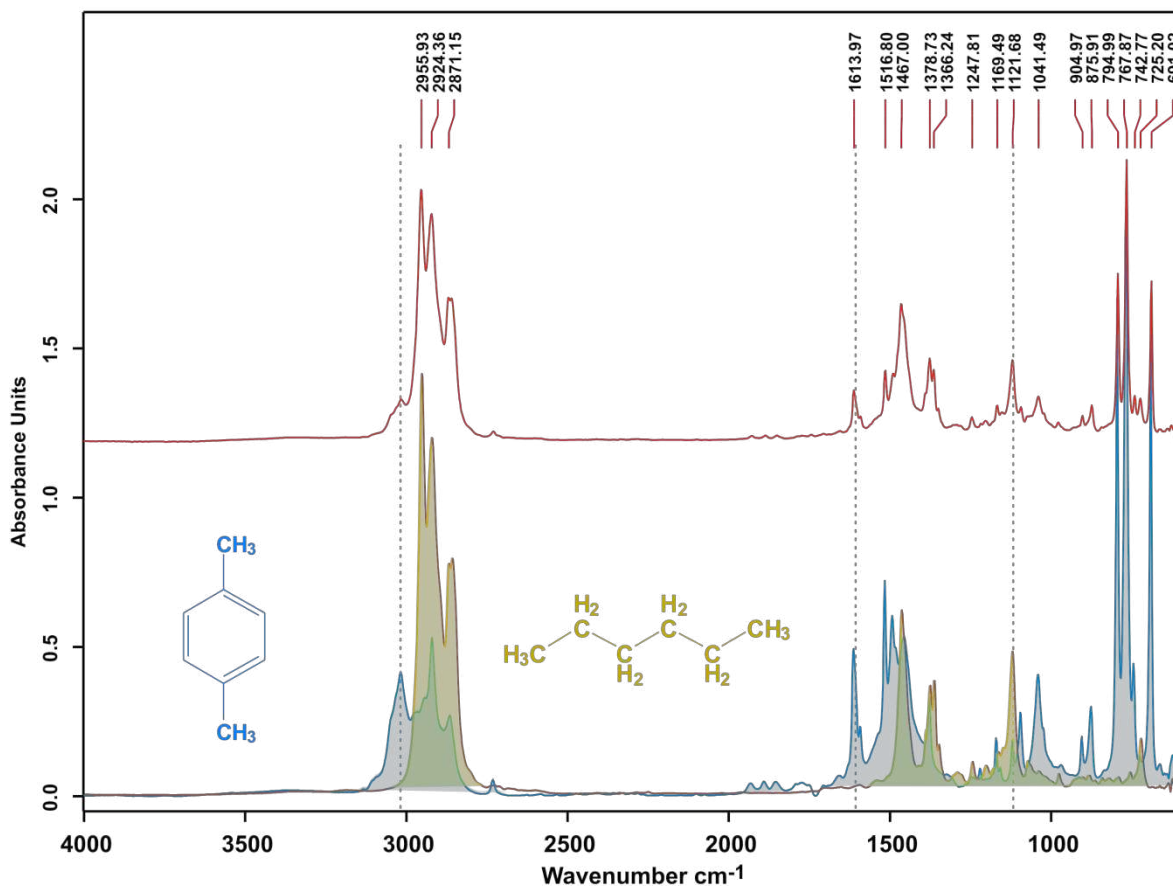


The major disadvantage to this method is losing the peak picking labels and not being able to highlight important peaks/data. While journals do not usually want peak picking wavenumber figure labels in the main body of a paper, they are very good for dissertations or supplementary material. Most plotting programs can handle stack and multiple plots, but fine tuning can be a pain.

**Plot Data as Postscript File, Import into Vector Drawing Program & Edit.** This method works equally well for any spectrum (IR, NMR, etc) that you can save as a postscript file. I have loaded CorelDraw, a vector drawing and editing program, onto both FT-IR computers. A student edition of the CorelDraw X3 program suite that includes PhotoPaint (photo & bitmap editing program) and CorelCapture (screen capturing utility) costs about \$110 at the bookstore. Adobe Illustrator is another good (but more expensive) vector drawing/editing program.

Below is an example of what you can do to “enhance” an FT-IR using one of these drawing programs. Time involved using CorelDraw? About 30 mins (once you know what you are doing).

### FT-IR of Xylene, Hexane, & Mixture



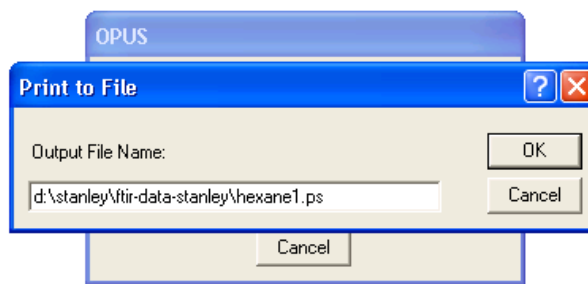
It is quite easy to color specific peaks by changing the line color, thickness, or, with a little more effort, using a background fill (solid or transparent) as shown above (or a mixture of both). ChemDraw structures are also easily added. This is also probably the easiest way to get a nice stacked plot of several spectra. You can easily rescale the intensity and position of the spectra to get a good view. See page 37 for more details on editing with CorelDraw.

**Why not use the Copy function or Print to Clipboard?** Copy and Print to clipboard are two options that capture a Windows MetaGraphic File (WMF), which is a vector graphics file of what you are seeing on the screen. The problem I have found with this and OPUS is that the resolution of what you capture seems to vary in a way that I don't fully understand with both operating system (Windows XP vs. Vista) and screen resolution. Sometimes you get a fairly high resolution capture of your spectrum, sometimes it looks terrible. Printing your spectrum to a postscript file and then importing that file into the drawing program produces a higher resolution spectrum curve consistently.

**Generating a Postscript File.** We have created postscript file printers for both of the FT-IR computers labeled **Postscript to File**. In OPUS go to **File** → **Print Setup** and select this as your printer and make sure the **Landscape mode** is chosen. See Prof. Stanley if you want to set this up on your lab or notebook computer, not all postscript drivers work equally well for this (we are using the Xerox Phaser 7400 postscript driver).

Then do a Quick Print of your spectrum (and peak picking if you want). **Only do one spectrum at a time!!** If you have multiple spectra displayed, un-display them by right clicking the trace (or ATR) display data block in the file window and select the “Remove from Display” option. This does NOT delete the spectrum, just changes its color so it doesn’t appear on the screen.

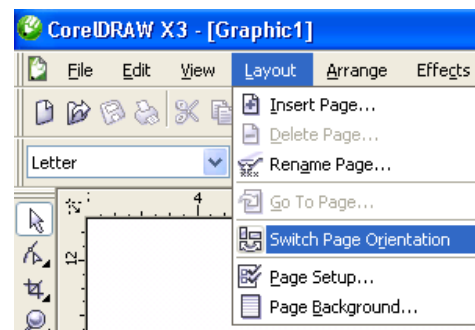
After clicking the Quick Print icon, a file save box will pop up similar to that shown to the right. You need to input the drive and directory where the file will be saved. This is NOT the standard file save dialog box that lets you select the directory to save the file, so it is important that you know the drive, your directory name, and in what subdirectory you want to save it. I suggest using **.ps** as your file extension – both Adobe Acrobat and CorelDraw automatically recognize this as a postscript file.



**Using CorelDraw to Enhance your Plot.** The procedure shown below will work for almost any postscript file of any spectrum (NMR, IR, mass spec, X-ray powder diffraction, etc). The following procedure does not go into excessive detail. Please see Prof. Stanley for more information on using CorelDraw if you have questions.

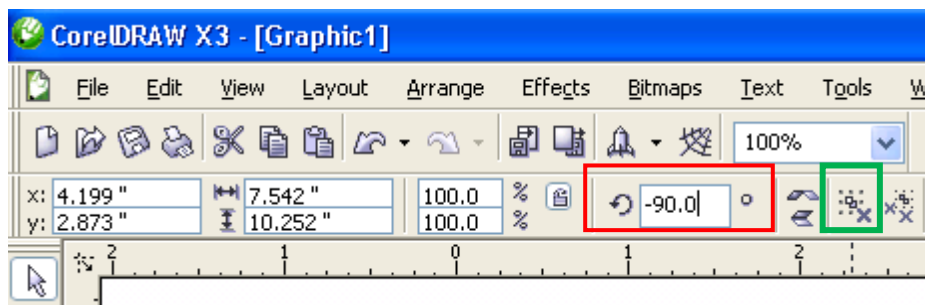


Start CorelDraw, select “**New**” from the welcome screen, and switch the page orientation into landscape mode by selecting **Layout**, then **Switch Page Orientation** from the menu that drops down (figure shown to right).



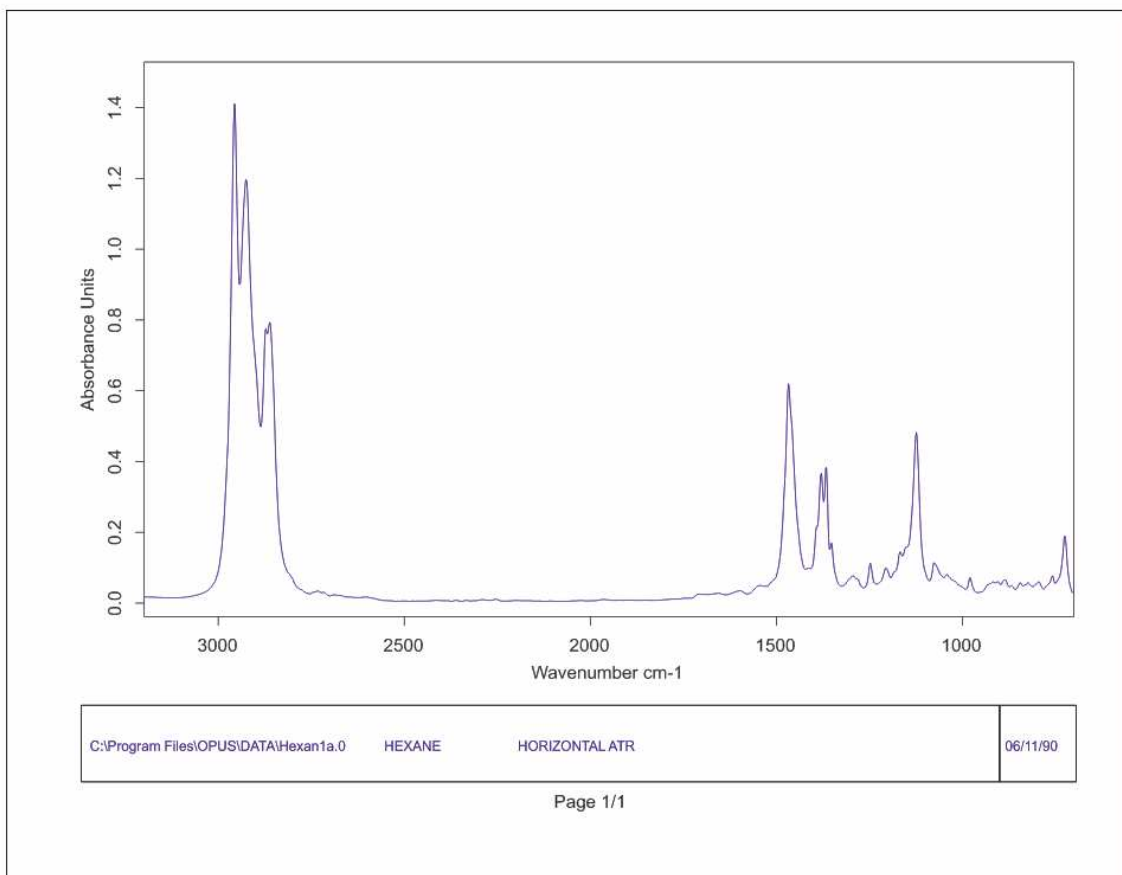
Select **File** → **Import** and select your Postscript file for import. If the file has a .ps extension CorelDraw should recognize it as a Postscript file and ask if you want to import text as text (fonts) or curves. Select text and next. A screen will then pop up with suggested font substitutions. Just click OK to proceed. The CorelDraw cursor will then change to a corner. You can now click on the screen to import the spectrum.

The spectrum will import in a vertical orientation, so you need to rotate it by  $-90.0^\circ$  to get it into the proper “horizontal” orientation. Use the Rotate menu box and type in  $-90.0^\circ$  (boxed in red below) and carriage return. Reposition the spectrum (click and drag) so it is oriented nicely in the page definition space (page outline).



The imported spectrum and associated text is all grouped together as one object. You need to **ungroup** the imported figure so you can edit the various sub-pieces (spectrum, fonts, colors, etc.). Click on the spectrum to select it, then click on the **ungroup** icon on the main menu, near the rotate box. This **ungroup** icon is boxed in green on the previous figure. Next to it is an **ungroup all** icon, which in this case will do the same thing.

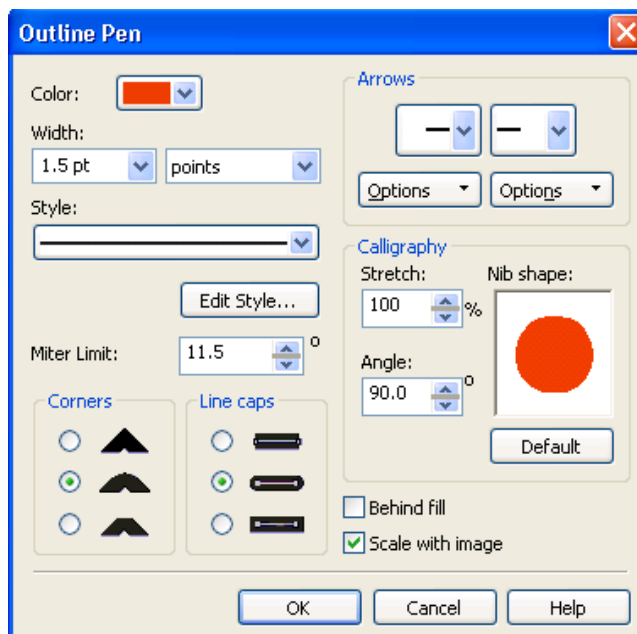
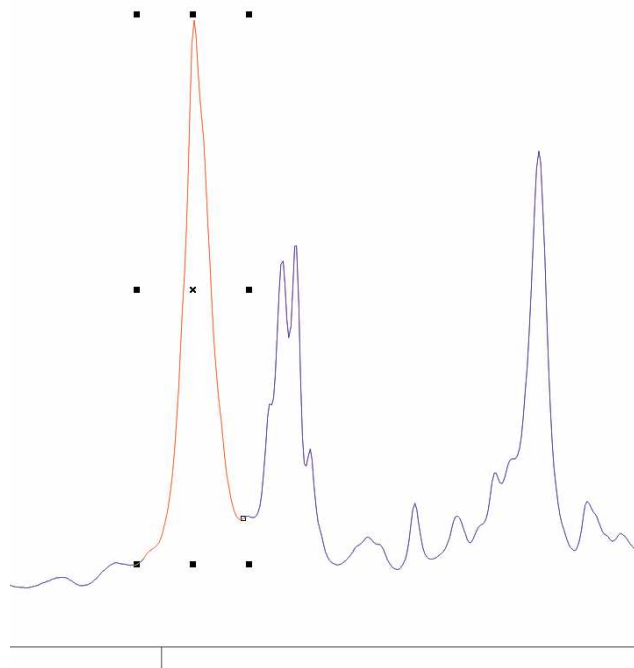
Click away from the spectrum to unselect it. Now you can click on any of the spectrum object to edit them. The main screen should look something like that shown below (I did not display peak picking on this spectrum, so they didn't print).



An important point at this juncture is to realize that the spectrum trace has also been un-grouped into a 1000+ separate data points connected by lines. If you click on the spectrum trace you will see that one data point is selected. Be careful that you do NOT move any of the data points! That would be considered modifying your data, which could be an ethical violation. It is OK to change the intensity of the entire spectrum so long as you are not trying to make quantitative comparisons between different samples/spectra.

At this point we can carefully work with the spectrum to select some peaks to highlight them by selectively re-coloring the line and/or changing the line thickness for emphasis. For example, I can use the mouse to click and select a box around one of the peaks to pick all the data points and lines comprising that peak. Right clicking on one of the colors on the right side color pallet (more available by clicking on the expand arrow at the bottom) will change the line color. Left mouse clicking changes fill or text/font colors. But at this point we have no closed objects so there is no fill color to change.

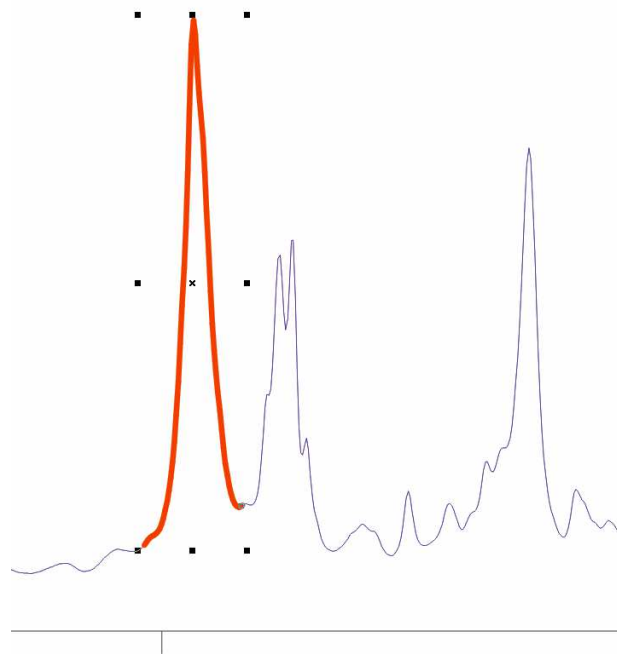
The figure below shows one of the peaks selected and the line color (right click) changed to red.



The line thickness can now be changed for this red line by clicking on the fountain pen nib (**Outline Tool**) on the left-hand vertical set of icons, then selecting the fountain pen nib icon again. This brings up the Outline Pen screen shown above. One can now change the line thickness to whatever value desired. I suggest clicking on the Scale with Image box as well. This will rescale the line thickness if you change the size of the image within CorelDraw. The figure to the right shows the effect of increasing the line thickness from the imported value of 0.199 points to 1.5 points.

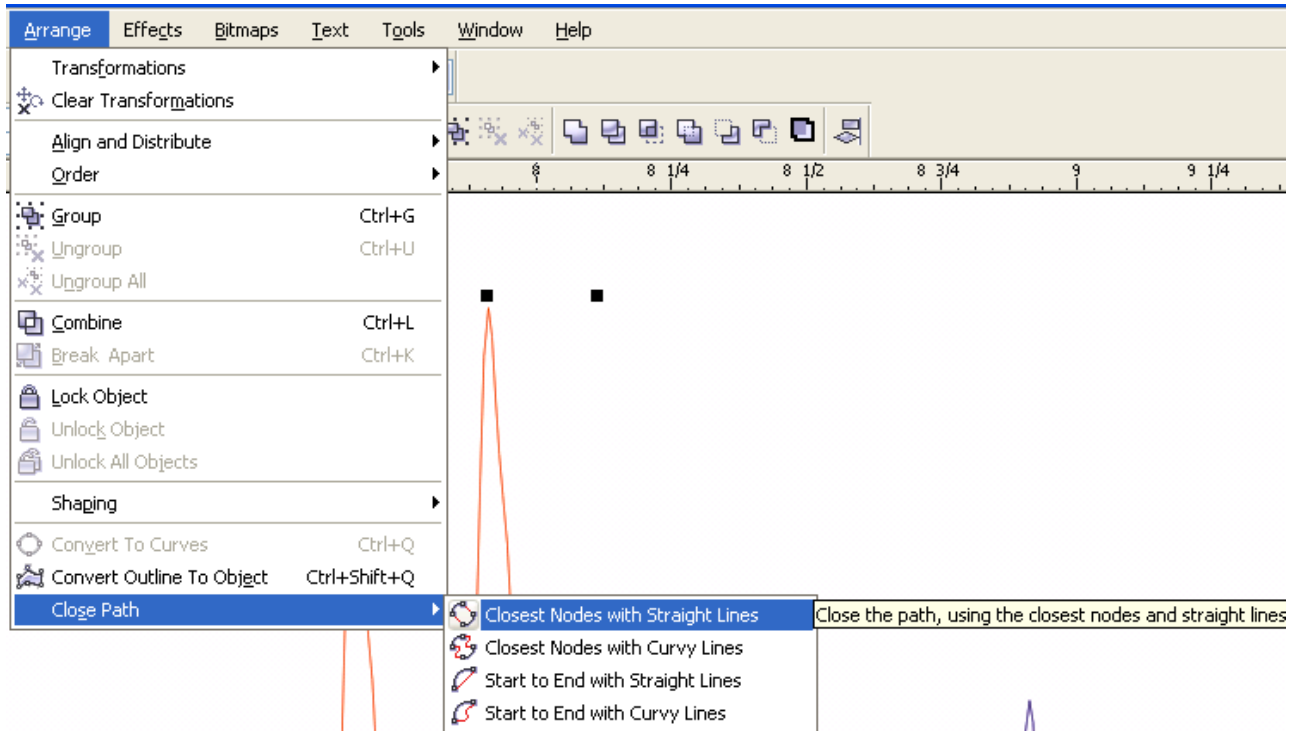
Generally, however, I think it is better to make the entire spectrum the same line thickness and highlight peaks of interest with different colors or using a fill color (discussed next).

To use a fill color you should copy (control-C) and paste (control-V) the selected peak to make another version to convert into a filled area. When you paste a copied object into CorelDraw it pastes it into the exact same position so it looks like nothing has been done!! But a new copy has been pasted and you can verify this by using the arrow keys to move the object a bit so you can see the new object. Alternatively you can do a control-D key sequence, which will duplicate the object and automatically translate it a bit so you can see the new duplicate more clearly.



**IMPORTANT Point:** Remember that your original spectrum trace is still composed of thousands of individual data points. So it is critically important that you move this new copied peak to a location where it is NOT overlapping with the original spectrum. If you click somewhere else on the page the new object (peak) will be deselected and if it is overlapping with the original spectrum trace you will not be able to select one or the other cleanly.

To this copied or duplicate peak you want to now click on the **Arrange** main menu category, then choose **Close Path** —> **Closest Nodes with Straight Lines** (next figure).

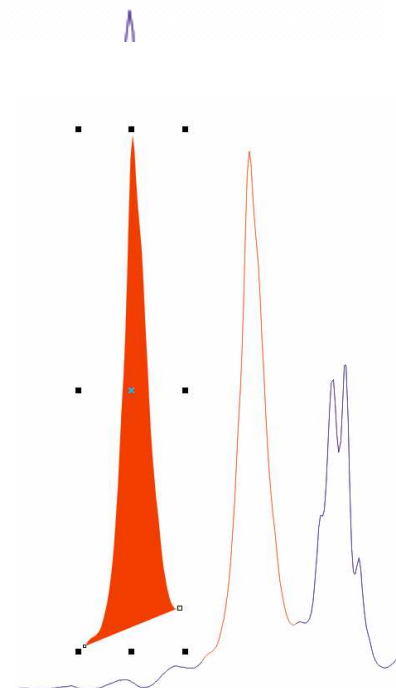
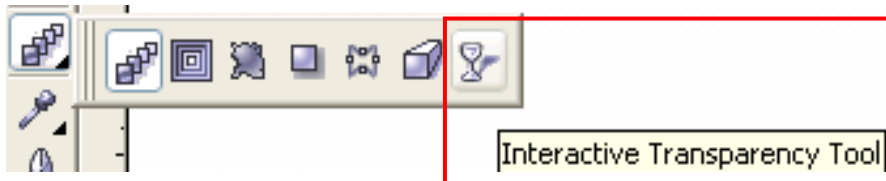


That generates the closed object shown to the right (shifted away from the original spectrum), which was initially hollow.

**This has now been converted into a single object with a closed shape.**

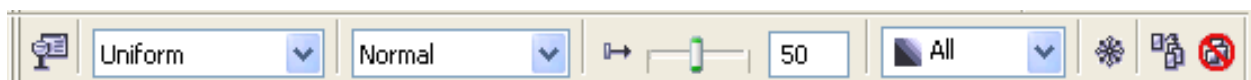
I then left clicked on the red color to fill it with red and right clicked on the no outline color (white box with an X through it at the top of the color scale) to remove the outline.

An easy way to get a lightly shaded color for this filled peak is to use the transparency tool/option, shown below.



Selecting the “cup” icon changes the main menu and creates a new group of icons & selections shown below.





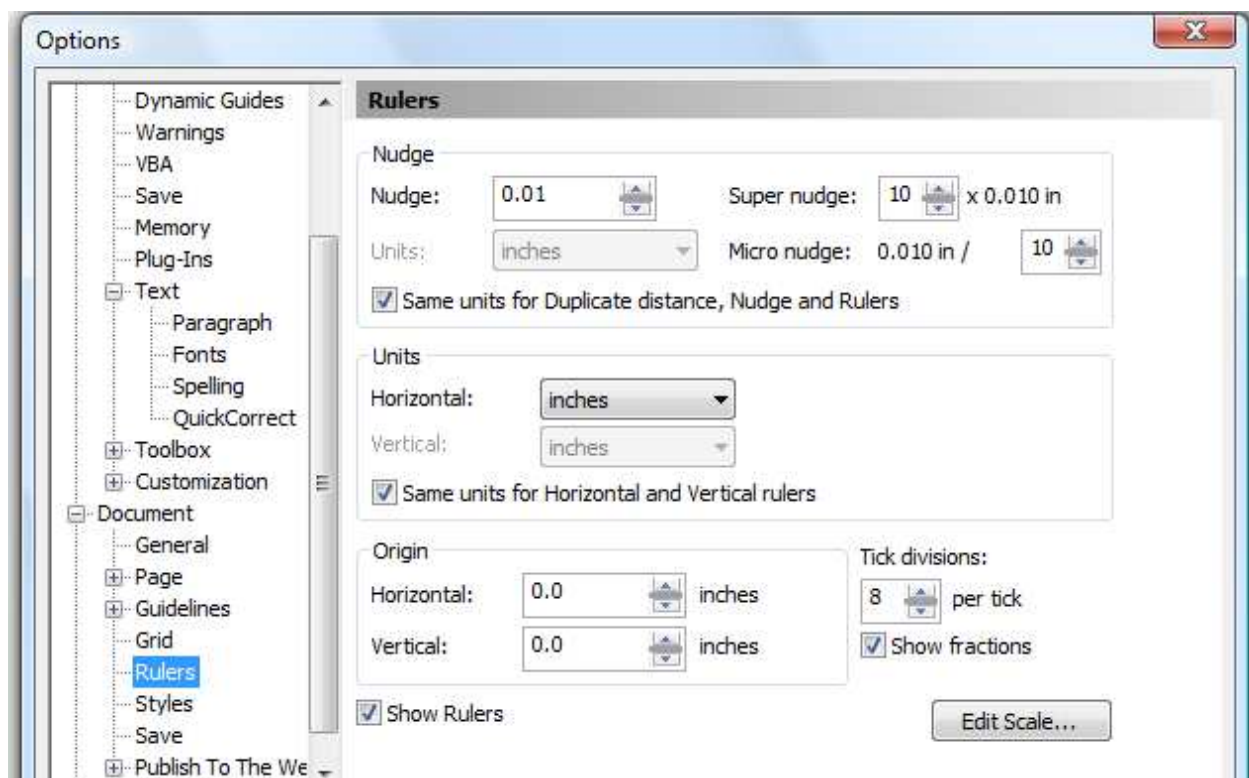
I changed the first box to “**Uniform**”, which activated the other items to the right of it. The box with **50** in it refers to the amount of transparency applied to the object. The higher the number the lighter and more transparent it is. There is a slider control to the left of this, but typing in a number is more accurate. Transparency not only lightens the color but allows objects below it to show through. As shown in the spectrum example on page 36 this can also be used to blend colors to show overlapping regions.

At this point I recommend moving the filled transparent peak off to the side and proceeding with any other peaks you want to fill and highlight.

Once you are done, you should carefully select the entire spectrum (watch out that you do NOT select any of the surrounding lines!) and group it together as a single object. If you are pretty sure that you do NOT want to do any more peak abstracting the other option is to select the entire spectrum and click on **Arrange** → **Combine**. This will convert all the separate points (nodes) into a connected curve (a single object). **BUT, the Combine command will NOT allow different line colors within a spectrum. So if you have changed the line color of some of the peaks use the Group function.**

Once the spectrum is grouped together or combined, you can move the filled peaks back and overlay them onto the appropriate areas on the spectrum. Then do another group command.

To get fine object movement control (needed to overlay your filled peaks on your original spectrum) using the arrow keys you should change the **Ruler Nudge** option under **Tools** → **Options** → **Document** → **Rulers**. Then change the **Nudge**, **Super nudge**, and **micro nudge** values as shown below. Holding down the control key while pressing an arrow key will move an object by micro amounts. Holding down the shift key while pressing an arrow key will move the object macro amounts. After changing these values, make them the default by clicking on **Tools** → **Save Settings as Default**.



**Text Editing:** Text is imported as text and can easily be selected and the fonts, size, and weight (italics) changed. I personally like **Arial bold** with a font size of about 14 for the wavenumber scale.

Superscripts and subscripts can be specified using the text edit command, BUT the super- and subscripts generated this way look way too small. I, therefore, recommend using the **Shape Tool** button (shown to the right) that is right below the select button on the left-hand side menu. Clicking on the text using the **Shape Tool** places small boxes to the left of each character and places two corner arrow objects, one pointing down and the other on the far right pointing to the left.



Wavefunction (cm<sup>-1</sup>)

The small empty squares to the left of each character allow them to be selected and individually modified and moved using the mouse or arrow keys. This allows you to fine tune the superscripting or subscript size and location (just make sure you are consistent). Holding down the shift key and left clicking the two small boxes for the -1 selects both characters for editing. I decreased the font size using the top menu Font boxes and then while they were still selected used the up arrow key to move them up and to the left a bit to give the following result:

Wavefunction (cm<sup>-1</sup>)

The arrow object to the far left will spread out (or condense) the spacing of the text. The down arrow at the front of the text will interactively change the line spacing between two or more lines of text.

You can also color and rotate text. The outline and fill colors for text can be different, which is good for light colored text (example below). The outline weight or heaviness can also be easily adjusted.

Wavefunction (cm<sup>-1</sup>)

**Multiple Spectra:** When importing multiple spectra to do a stack or overlay plot, import each in separately and make sure you edit that trace away from the others and then group it together as one object once you are done. Also make sure that each is on the same wavenumber scale!!

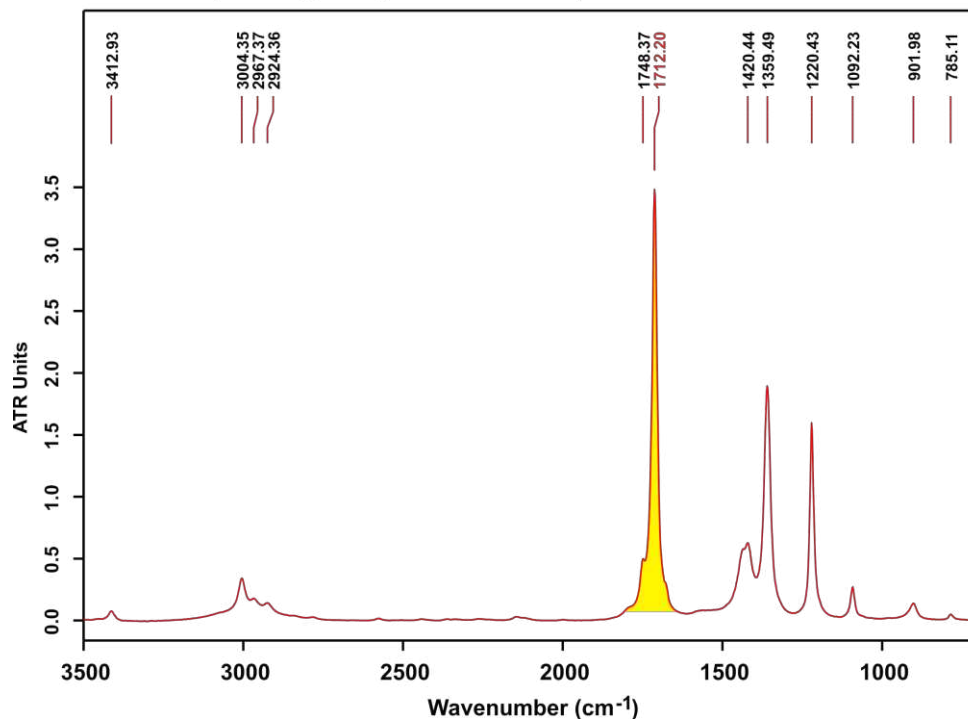
The reason that you can't import in an overlaid set of traces all at once is that there is almost no way to select one trace for modification when they are overlapping in any significant way. Once you ungroup the traces each data point is separate. If you are happy with the colors and do not want to do anything fancy, it is easy to select all the traces and change their line weights without affecting their colors. But you won't be able to move one trace relative to the others.

I do not know why OPUS won't print or export stacked plots well, hopefully the new version (6.5) will handle this better.

**Rescaling Spectra:** Be careful rescaling spectra in CorelDraw. If you change the vertical scale remember that the absorbance or transmittance scale will no longer be accurate (unless you scale that as well). This is especially important for the horizontal scale & axis. If you want to show an expanded portion of your spectrum with a wavenumber scale, I suggest expanding it in OPUS, printing to a separate Postscript file, then importing into CorelDraw as a separate spectrum that you edit, group, and position on your main spectrum as an insert.

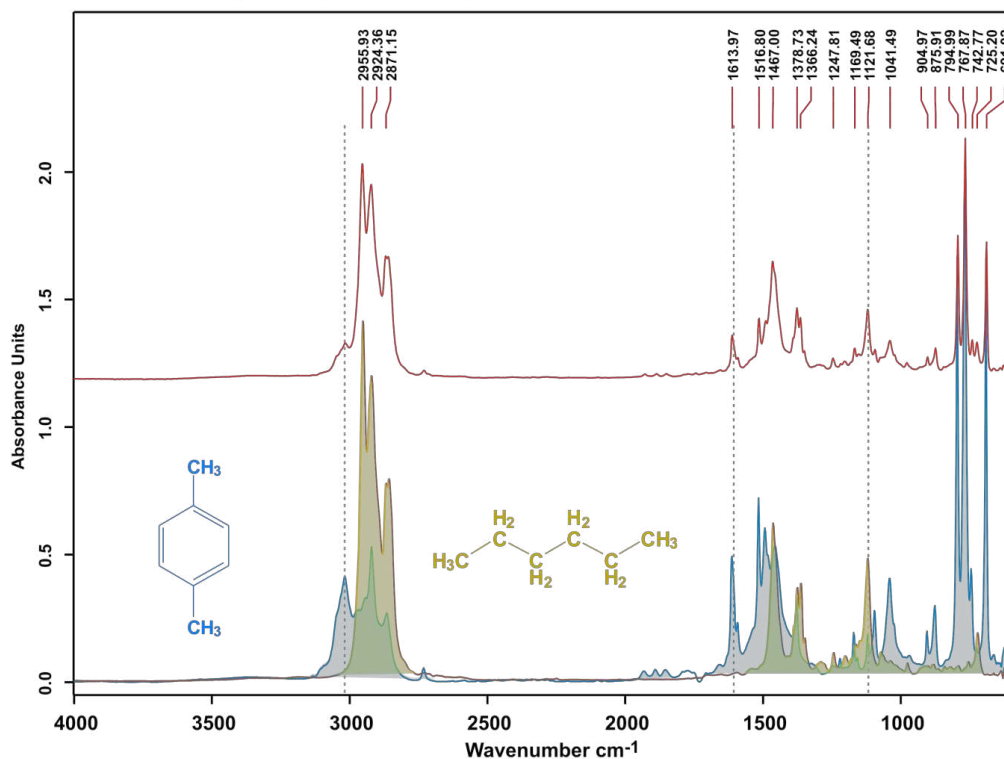
**Two Examples:** A simple single ATR absorption spectrum of acetone printed from OPUS as a Postscript file, imported into CorelDraw, and “fixed up” is shown below. Time involved: 10 mins (includes a phone call “interruption”).

### Acetone (wash grade, Pike ATR cell)



The “fancy” overlaid and stack plot shown earlier. This took about 30 mins (including the ChemDrawings).

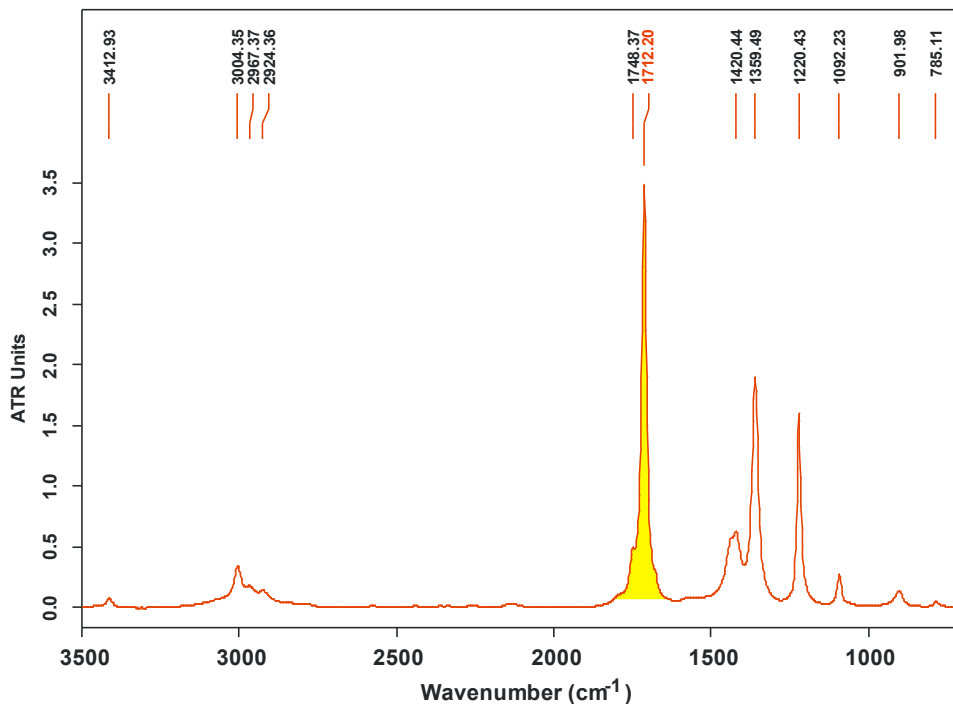
### FT-IR of Xylene, Hexane, & Mixture



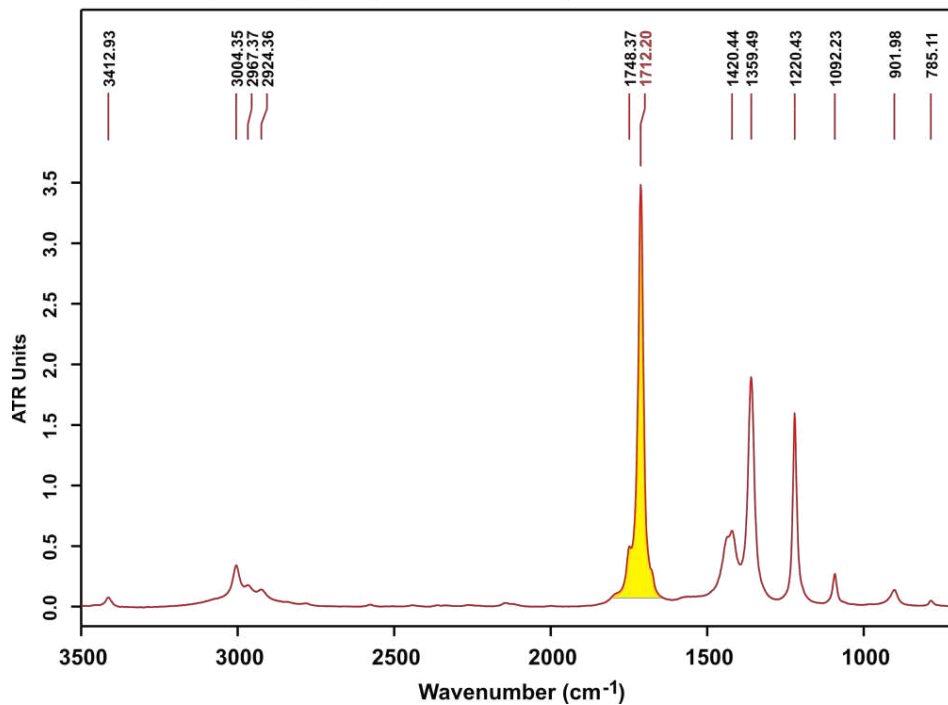
**Exporting from CorelDraw to Word or PowerPoint:** Although you can sometimes copy and paste from CorelDraw directly to Word (or other Office apps), it is better if you export your spectrum or drawing as a Portable Network Graphics (PNG) file and import that into Word or Powerpoint. Although a PNG file is a bitmap it has some very nice features such as anti-aliasing (to smooth lines), compact size (for a bitmap), and 24 bit color depth. TIFF bitmaps are much larger than PNG bitmaps.

Shown below is the acetone spectrum directly copied and pasted from CorelDraw into this Word document with the exported PNG spectrum file right below. Which one do you think is better?

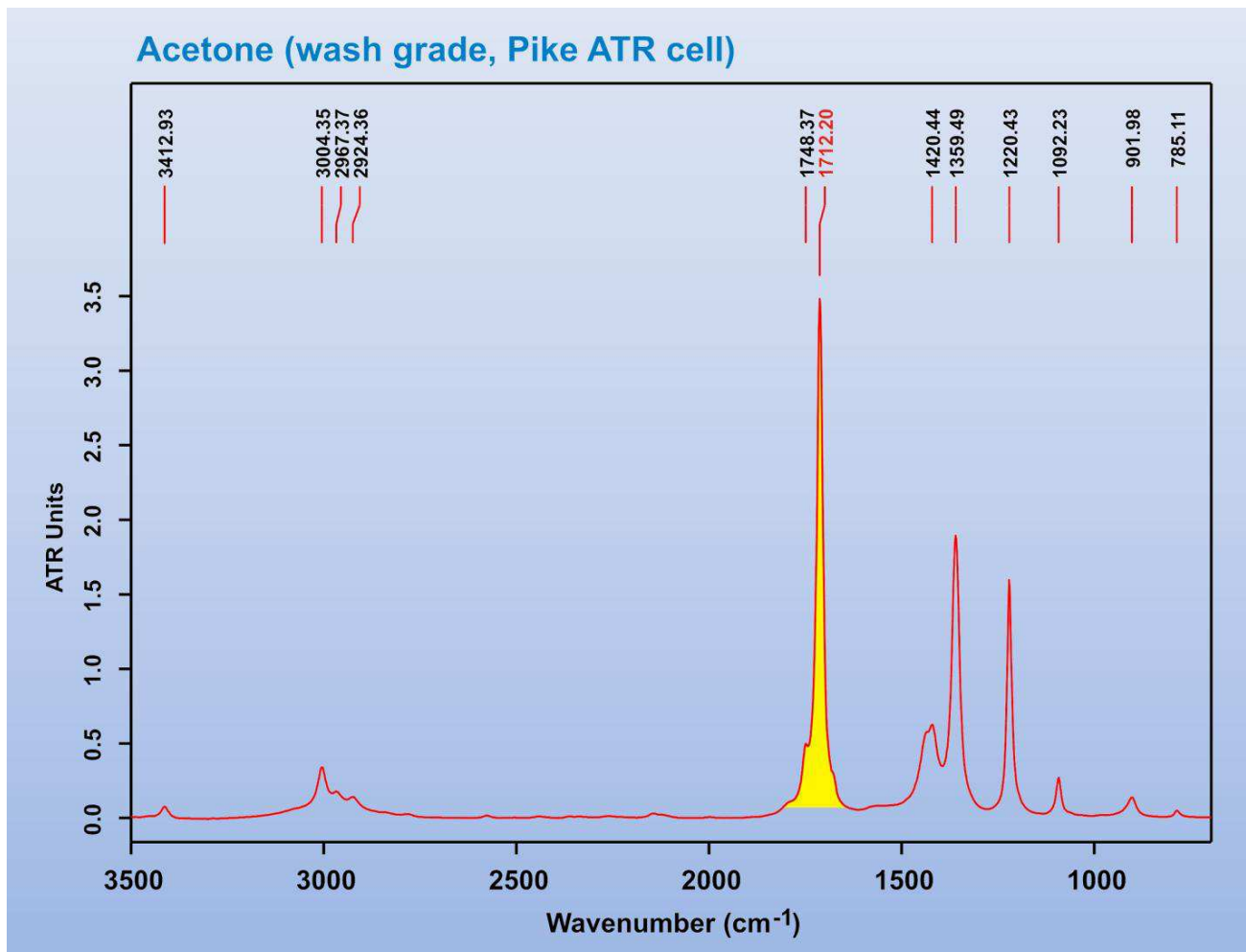
**Acetone (wash grade, Pike ATR cell)**



**Acetone (wash grade, Pike ATR cell)**



The other BIG advantage of using a PNG graphics file of your spectrum or drawing is that it looks great in PowerPoint due to the anti-aliasing effect that smoothes out lines, removing the “jaggy” look. Yet another potential advantage is that the PNG graphic can be created with a transparent background. This is great if you have a lightly colored background on your PowerPoint slides that you want to show through the image. A screen capture of a PowerPoint slide with a light blue gradated background and the PNG graphics file of the acetone spectrum is shown below.



Of course, you may not always want a transparent background and that is one of the options when you export the PNG file from CorelDraw (along with resolution, color depth, etc).

One “problem” with PNG graphic files is that Adobe Acrobat does not generally do a good job of converting these into the PDF file format. However, the PDF file export option in the Microsoft Office 2007 suite does an excellent job of translating PNG graphics files to PDF. Note that if you are using Office 2007 the PDF file creator is not initially present (due to a lawsuit by Adobe). But you can easily download and install the PDF file creation module for Office from their web site. I strongly recommend doing this. The Office PDF file creation function is accessed under **File** → **Save As** → **PDF or XPS**. This PDF file was generated using this Office feature.

## Copying and Backing up Your Data

You can use a USB flash drive or external hard disk drive to copy and backup all your data. I have put a USB extension cord attached to the front of the Dell computer to make it easier to plug in your storage device. Use Windows Explorer to copy your entire directory over to your storage device, or just the files in question.

**Network file transfers.** You can also directly link into the FT-IR computer and transfer files over the network to your lab computer (or notebook computer). The procedure varies depending on whether you are on campus or not (on campus is easiest) and the computer/operating system. The instructions below are for Windows XP or Vista computers. I don't know much about Mac's, for help see Les Butler or our computer support personnel.

The FT-IR Computer in Choppin 702 has two names assigned to it. The internal Windows netbios name is **CH702-FTIR**. The automatic LSU network (netreg) registration name tied to your PAWS ID is **gstanley-7.lsu.edu**. These will change if we shift the computer to the LSU Active Directory.

**On-Campus Access.** If you are accessing the computer disk drive from on campus you should use the CH702-FTIR name (not case sensitive).

### Windows XP:

- 1) Go to **Start** → **My Network Places** and click on **Add a network place**. This starts the add network place wizard. Click next to continue.
- 2) Select **"Choose another network location"**, click next.
- 3) Type in the name of the computer in question. In this case it will be:  
**\\ch702-ftir\d\user-directory-name**  
Note that the computer must have file and printer sharing enabled and disk drive sharing setup (**d** in this case).
- 4) You should now get a Windows Login screen. You need to type in your username for that computer and the password. Click on "remember my password" if you want to by-pass this screen in the future (if you change your password this login screen will reappear).
- 5) If the username and password is correct you will now get a screen with a suggested name for this new network place. Modify the name to be clear and informative to you. Click next and then Finish. This will open by default a file view window to the drive/folder in question and create a new Network Place link/shortcut under Network Places. You can copy this to your desktop if you intend on accessing it a lot.

### Windows Vista:

- 1) Go to **Start** → **Computer**, this opens up the screen showing all the disk drives and network paths to other computers.
- 2) Right mouse click on the blank area of the screen in the main window. Select **"Add Network Location"** from the menu that appears. This initiates the **Add Network Location** wizard.
- 3) Choose **"Custom Network Location"** and type in the name of the computer in question. In this case it will be:  
**\\ch702-ftir\d\user-directory-name**  
Just as for Windows XP, the computer you are linking into (and yours!) must have file and printer sharing enabled and disk drive sharing setup (**d** in this case).

- 4) You should now get a Windows Login screen. You need to type in your username for that computer and the password. Click on “remember my password” if you want to by-pass this screen in the future (if you change your password this login screen will reappear).
- 5) If the username and password is correct you will now get a screen with a suggested name for this new network place. Modify the name to be clear and informative to you. Click next and then Finish. This will open by default a file view window to the drive/folder in question and create a new Network Location link/shortcut under Computer. You can copy this to your desktop if you intend on accessing it a lot.

Adding a Network Place or Location is analogous to Mapping a Network Drive. The advantage to defining a Network Place in Windows XP is that the password appears to be firmly stored. When you Map a network drive you assign an open drive letter, but my XP computer often forgot the account password needed to re-link into the drive mapped computer on a reboot. Vista is much better about remembering passwords for Mapped Network Drives, so there is little difference between the two.

Both mapped network drives and network locations appear under Computer in Windows Vista. For Windows XP network places are listed under My Network Places (usually below My Computer in the file explorer listing). Once you have these defined you can readily copy files back and forth between the two computers.

**Off-Campus Access.** If you are accessing the computer from OFF-campus the easiest way to access your data is to use the **Remote Desktop** program and the [gstanley-7.lsu.edu](http://gstanley-7.lsu.edu) computer name (Windows XP or Vista). For those of you that have not used Remote Desktop it is a very powerful program that allows you to login to a remote computer and work on that computer and have access to all its resources, files, and programs as if you are actually there working on the computer. It works through the Windows Firewall, but you need to setup the remote computer to accept Remote Desktop sessions (Remote tab located under My Computer properties in Windows XP, or Computer, then System Properties, Remote tab in Windows Vista).

When establishing a **Remote Desktop** link to a computer you need to specify via the **Options** selection (at least the first time you setup the connection) that you want to share resources between the computers and the type of internet connection being used (the **Experience** tab). Sharing drives is the default option and allows one to copy files between both computers.

**WARNING:** a Windows computer can only have one user logged in at a time, either directly or via Remote Desktop. When you login to a computer using Remote Desktop you will get a message if another user is logged in and a warning that if you proceed you will abort that users session and kick them off. If you are the one logged in locally and never logged off, that is fine. But if someone else is logged in you do NOT want to kick them off unless you have their permission or know that they are not actively using the computer (i.e., they forgot to logoff and are not doing anything on the computer). You also have to have administrator privileges to do this (I believe). **When you are done, remember to LOGOUT of your account.** Remote Desktop will then automatically close. If you simply exit from the Remote Desktop program you will still be logged onto the computer in question and any programs will continue running.

The more complicated way to access the files on a campus Windows computer from off-campus is to establish a virtual private network (VPN) connection. IF the campus computer in question is either on Active Directory -OR- part of the LSU workgroup you can then map a drive or setup a network place/location. LSU has upgraded their network security to NOT allow Windows File and Printer sharing functions (drive mapping, network places) from off-campus except via a VPN connection and to computers that are part of the Active Directory or LSU workgroup. See Prof. Stanley or the computer support personnel (Greg O'Dell, Bob Zinn) for more information on this.