## Tensor 37 FTIR Operating Instructions
*Revised 12/19/2014 S.V.*

**Location:** **1238 Hach Hall**
**Contact:** **Steve Veysey, 1234 Hach Hall**

### Safety

All researchers working in 1238 Hach Hall must complete the EH&S course: *"Fire Safety and Extinguisher Training"*. When preparing samples in this room, please wear all appropriate personal protective equipment. Aprons, safety glasses, and rubber gloves are available in 1238A Hach Hall. If solvents are involved, please prepare your sample in room 1238A.

Properly dispose of glass pipettes and plastic sharps in the containers provided. Waste solvents can be disposed of in the waste containers provided in 1238A. All of the computers in this lab have direct links from the desktop to MSDS sheets, the EH&S Laboratory Safety Manual and to the CIF Safety Manual.

### Introduction

The Bruker Tensor 37 FT-IR is a versatile research grade instrument capable of measuring in the mid-IR region of the spectrum. The instrument is primarily configured for vapor phase analysis of components evolved from the attached Netzsch TGA/DSC system, but can also be used "stand alone" for normal measurements.

**Fig.1 Tensor 37 FTIR with TGA-IR Attachment**

**Overview**

The Tensor 37 is controlled by a Windows 7 PC communicating via a dedicated Ethernet cable to the spectrometer. The instrument optics and sample compartment are each continually purged with dry nitrogen. The instrument is always "on"; there are no hardware controls or switches on the spectrometer that require user attention.

Sample and reference cells are placed sequentially into the sample compartment as required. You may use the sample holders in the lab if they are suitable for your measurement. Salt plates, crystals, liquid cells, et cetera must be provided by the user. It takes approximately 10 minutes for 90% of the room air moisture and $CO_2$ to be purged from the compartment. For routine measurements there is no need to delay more than a minute or two, but for critical measurements consider purging at least ten minutes. Be sure to insert your holder completely, and to immediately replace the sample compartment lid.

**Fig. 2 Sample Compartment Access**
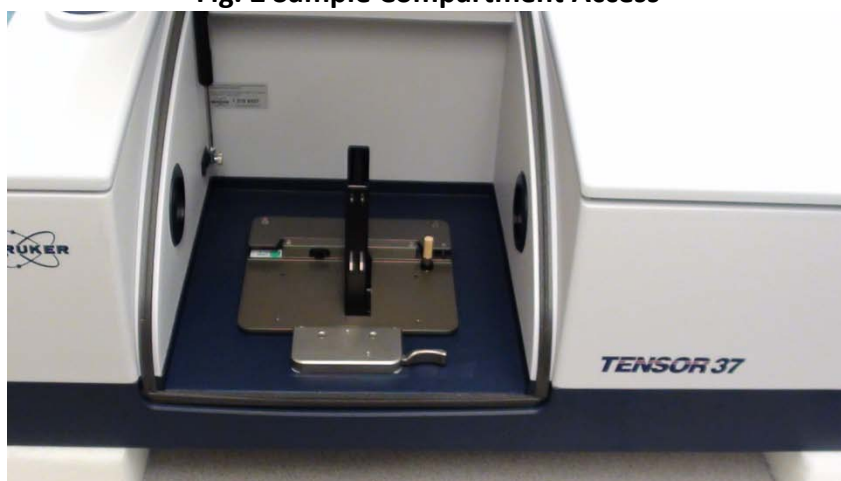


FTIR uses "single-beam" measurements in either transmission or reflectance mode to generate transmittance or absorbance spectra. The simplest measurement requires only two single-beam curves, one for the reference, and one for the sample. In this case, the final transmittance spectrum would just be the ratio (division) of the sample file to the reference file. Since absorbance is the negative log of transmittance, spectra viewed initially as Tr can be converted to the Ab view (and vice versa) without having to reacquire single-beam sample and single-beam reference files.

For critical work, consider acquiring three "single-beam" measurements. The first file would be your reference ratioed to the instrument background (empty channel); the second file would be your sample ratioed to the same instrument background file. The spectral subtraction features of the software would then be used to generate the most correct final spectrum of your sample.

**Sample Preparation**

The quality of an infrared spectrum is heavily dependent upon sample preparation. There are many possible ways to prepare your sample. This is a choice you must make based upon the nature of your sample and the research question you are trying to answer by acquiring infrared spectra. We offer a number of sample holders you may use once your sample is prepared.

**Fig. 3 Various Sample Holders**
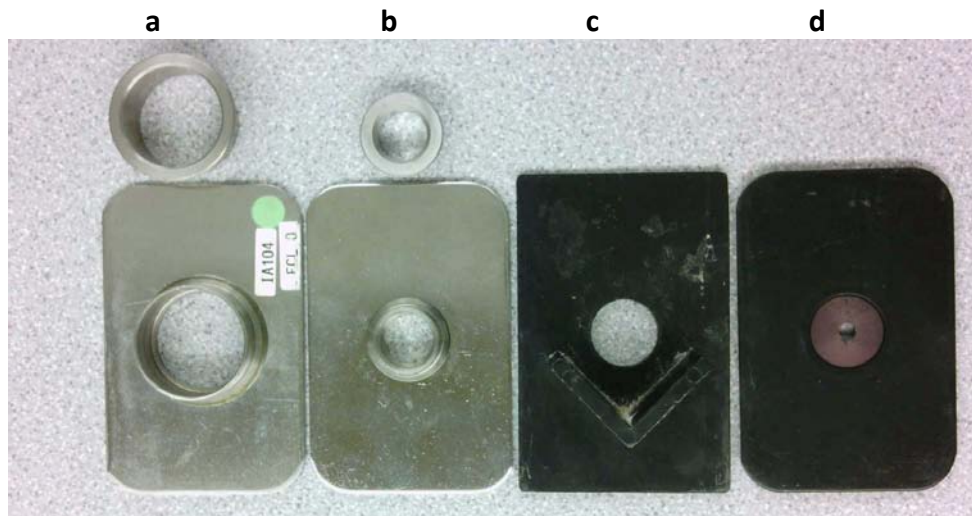


a. Used to hold 25mm single salt plates or two sandwiched salt plates.
b. Used to hold 12.5mm KBr pellets
c. Used to hold salt plates or the student KBr pellet assembly (7.5mm pellets)
d. Various aperture holders allow smaller samples to be mounted using tape.

*NaCL Salt Plates and IR Cards*

One of the easiest sample preparation methods involves dissolving a bit of your solid sample in a solvent (other than water or DMSO!) and spotting a drop or two onto a NaCl salt plate. When the solvent evaporates a thin film of your sample will normally be centered on the salt plate. If your sample is already a viscous liquid or paste, just smear a bit on the salt plate and form a thin layer by pressing with a straight-edge or another salt plate. Unpolished NaCl salt plates can be purchased from Chem Stores for $15-20. We provide a polishing kit in the lab for your use, and also a grinding kit should your salt plates eventually become pitted or wavy. See Figure 6.

**Fig. 4  IR Card and NaCL Salt Plate**

IR cards consist of a thin stretched plastic mounted in a cardboard or metal frame.  The plastic is generally either Teflon (C2F4 polymer) or polyethylene (C2H4 polymer). A drop of solution (or liquid) is placed directly on the plastic; as long as the plastic appears to "wet", you should get a reasonable spectrum.  There will be strong absorption bands from the CH2-stretch or the CF2-stretch creating a spectral dead-zone in those regions.  These cards can be purchased from various on-line sources.

*KBr Pellets*

A solid-solid mixture of 1% sample and 99% spectral grade KBr can be pressed into a glass-like pellet.  The powder mixture is then sandwiched between two steel dies with highly polished surfaces.  Pressure is applied to form the pellet.  We have a simple die-set available for student use.  The pellet formed is small (7.5mm) and cannot be extracted from the die assembly; it is run in-place.  Also shown is a more expensive version used to form removable 12.5mm pellets.  This is not available for student use.

**Fig. 5  Routine 7.5mm Student Die set and Research 12.5mm Die Set**
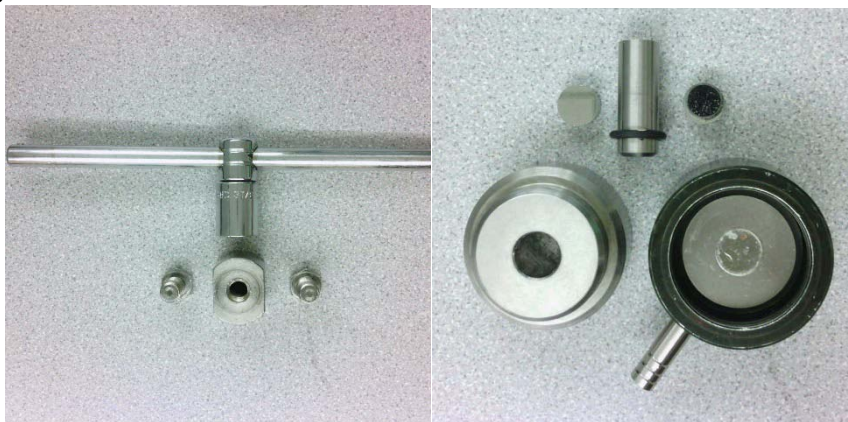


A hydraulic jack suitable for use with the more expensive styles of research quality KBr die sets is located on the table in 1240 Hach Hall.  You are required to provide your own die set.

**Fig. 6  Hydraulic Jack and Crystal Polishing Kit Located in the Lab**

_Liquid Cells_

These are not provided.  Liquid cells tend to be specific to the project requirements.  The proper crystal material and pathlength must be chosen based upon your needs.  Generally, one set of liquid cell support hardware can be used with a variety of sealed (fixed pathlength) cells, or demountable cells that allow pathlengths to be adjusted by using different spacers.  For aqueous use, demountable "minicells" with AgCl windows are often used.

**Fig. 7  Example of a Fixed Pathlegth Liquid Cell**



_Reflectance Measurements_

We have a variety of older diffuse reflectance, specular reflectance, and attenuated total reflectance (ATR) devices previously used with other FTIR's we have had.  These can be made to work in the Tensor 37 through the addition of the proper baseplate or mounting hardware.  This will require advance notice and you will be billed at $25.60 per hour for the time it takes staff to mount and align the attachment.  For the ATR assembly, you must purchase the appropriate crystal.  These expensive crystals are easily damaged and are not usually shared.

**Fig. 8 A Variety of Diffuse and Specular Reflectance and ATR Assemblies**
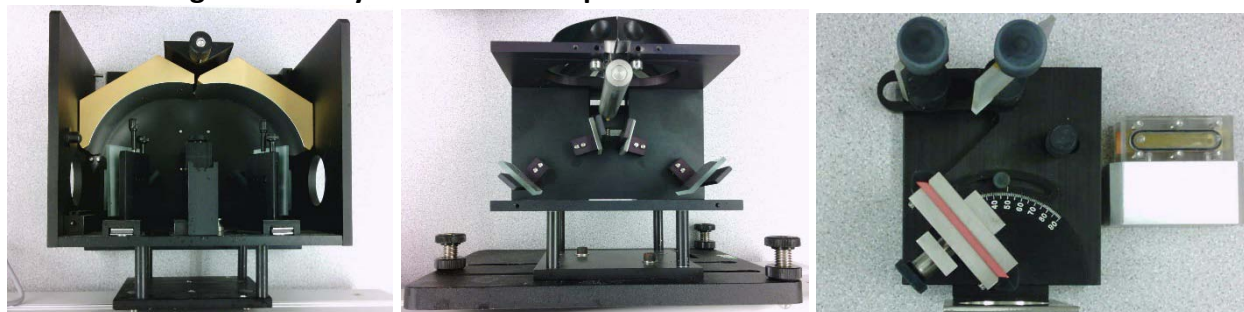


For more information about IR spectroscopy, measurement choices, and sample preparation methods, click on the Bruker IR Introduction shortcut on the desktop.

**Getting Started**

The computer should always be logged on as the Windows 7 Administrator account.  If it is necessary to log into Windows 7, do so as *username: Administrator, password: netzsch.*

Each registered user has been given directory space on the D: drive of the FTIR instrument control computer.  Your username is the directory name.  IR data files are automatically acquired in a system "Meas" directory on the C:\OPUS_7.2.139.1294\ path, and should remain there.  Copies or modified files can be stored in your space on the D: drive.

The FTIR software is called "OPUS".

Double click the OPUS icon located on the desktop.  This will bring up the OPUS Login view.  You are <u>not</u> assigned an individual account within OPUS.  Select the *MIR_Routine* workspace, then log in as *Administrator*.  The password is "OPUS".



The license manager view will pop up.  Select OK.



An instrument status dialog may appear. The 60139 status message can be ignored.  Close the status box.  Other messages could indicate a problem.
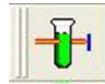
**Program View**

The program will now open to display a number of views and tools.
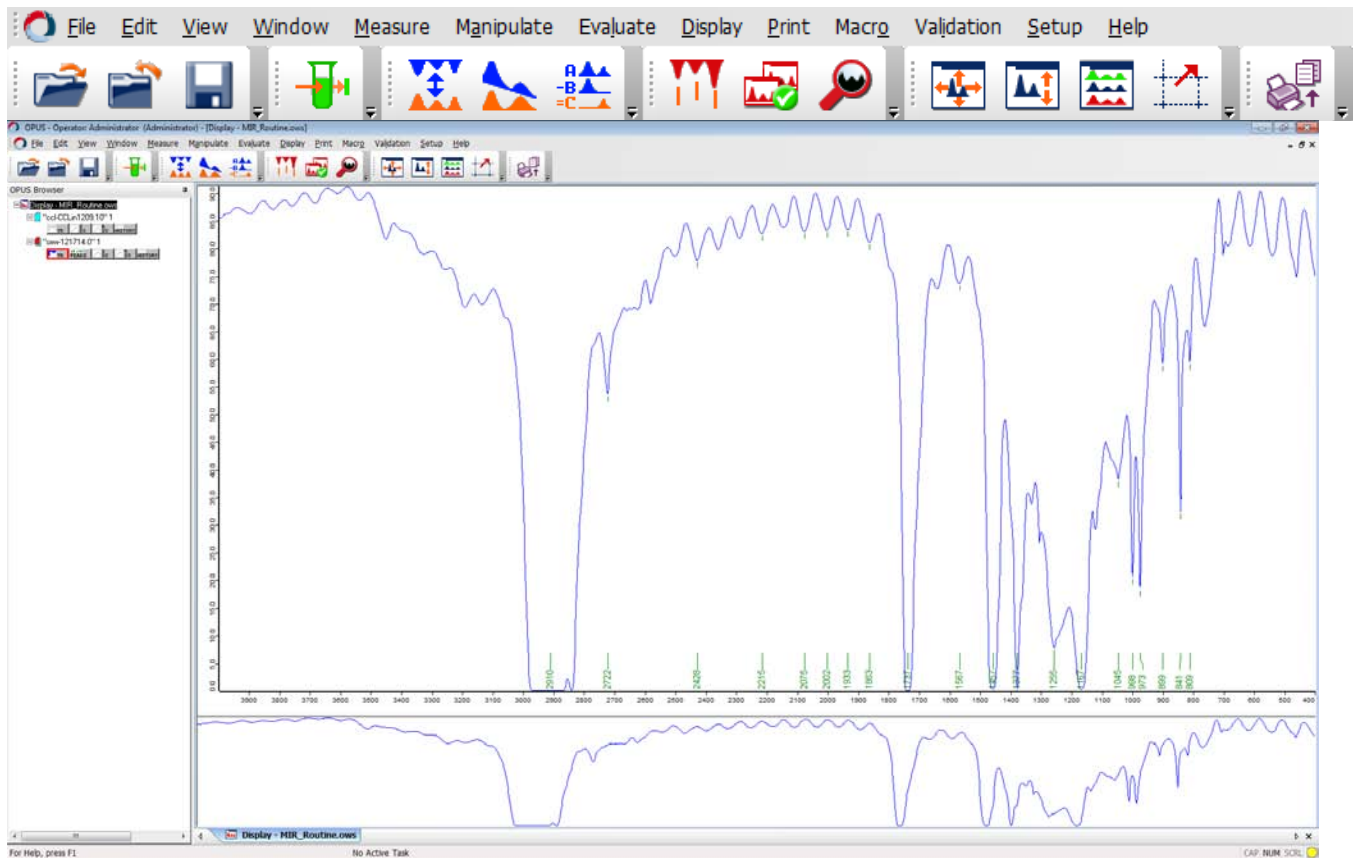


Routine MID-IR data acquisition and processing can be accomplished using just the icons on the tool bar.  Each icon's function can be displayed by placing the mouse over the button.

**Acquiring Data**

Use the <Measure> button to acquire new data.    This will open a multi-tab Measurement view.  It is only necessary to work with the *Basic* tab, but you may also modify some of the elements on the *Advanced* tab for use during your session.  Do not modify any elements on any of the other six tabs!  *Do not <save> any changes made on the Advanced tab!*



**Measurement - Basic Tab**

Parameter sets that have been modified from the proper default values will be highlighted in yellow. Always start by ⬛Load⬛ -ing a fresh copy of the experimental parameter file. Select *MIR_TR.XPM*, then select ⬛Open⬛.
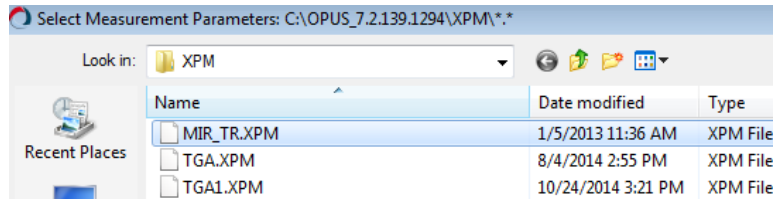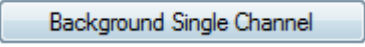


Once returned to the Measurement view you will note that the experiment name will no longer be highlighted in yellow. Do the following:
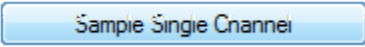
| | | |
|---|---|---|
| Verify - | Experiment: | MID_TR.XPM |
| | Operator Name: | Administrator |
| Modify - | Sample Description: | *<type in assigned initials (eg. abc-). Leave the rest alone!* |
| | Sample Form: | *<type in any text of your choosing>* |

This workspace is set up so that the filename automatically assigned to your measurement will consist of the text entered in the "Sample Description" field and mm/dd/yy date codes. Enter "abc-" in the sample description field, where abc- is your assigned three-letter code. ***Don't modify anything else on that line!*** If you make a mistake on this line, just reload a fresh copy of the experiment parameter file and start over.

During your training you will also be shown how to use the "*Measurement - Check Signal*" tab to verify instrument performance. It is not necessary to do this, but it can be used to quickly evaluating the quality of your sample preparation. Your interferogram center-burst amplitude should be greater than 22,000 counts with nothing in the sample compartment. Great sample prep would probably show an amplitude of about 10,000 counts; you will still get workable spectra if the sample is transmitting at least 1,000 counts.
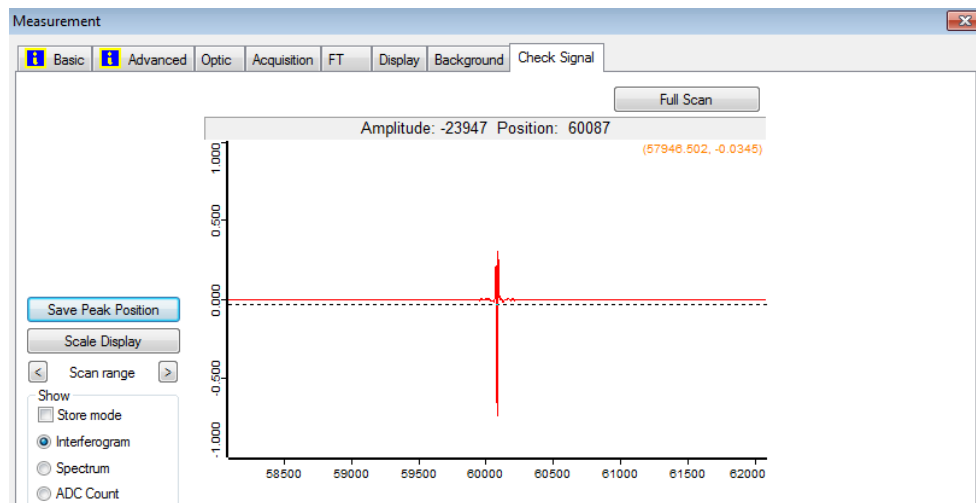
To acquire data, you must be viewing the *"Measurement – Basic"* tab.  After placing your reference sample into the purge box, press [Background Single Channel] to acquire the reference file.  Acquisition progress is displayed on the Task Bar.

Next, place your sample into the purge box and use [Sample Single Channel] to acquire the sample file.  After the file has been acquired, the resultant transmittance spectrum is displayed in both the Spectrum Window and Overview Window of the display.

**Processing Data**

The current spectrum should now be displayed in the Spectrum and Overview windows.  You may also retrieve previously acquired data stored on the hard disk by using the <Load> button.



*NOTE: Select < browser> from the View menu if the OPUS Browser frame is missing.*

You should become familiar with the use of this browser frame if you plan to process previously acquired data, or view more than one data file at a time.  Each file in the browser will consist of several "data blocks".  During your training you will learn the significance of each type of data block.  Any data block can be dragged onto the spectral display, or selected and removed from the display.
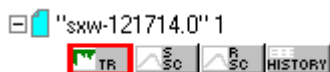


1.  Adjusting the Display

The spectral display-limits may be adjusted from either the Overview window or the Spectrum window.  In the Overview window, use the left mouse button to zoom in the X or Y direction by grabbing the edges of the display.
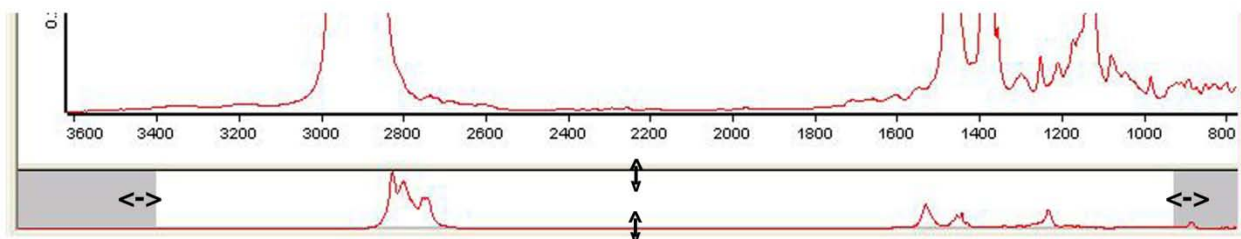


Four buttons on the Icon tool bar can be used to move between various display features:



*Unzoom; Zoom-Y; Stack; Zoom*

To adjust the display in the Spectrum window, select  to activate the zoom-in crosshairs. While holding the left mouse button, drag to form the desired expansion box. Click the left mouse button twice to create the expanded display.

2. Baseline Correction

Sloped baselines are usually caused by poor sample preparation. Baselines should be corrected before attempting to "Peak Pick". Click on  to enter the Baseline Correction view. On the *Select Files* tab, choose the appropriate file. If the wrong file is displayed, use the keyboard "delete" key to remove it from the tab view; then use your mouse to drag the correct file data block from the browser pane into the tab view.

On the *Select Method* tab, verify that the "scattering" correction using 64 baseline points has been selected. Click on <Correct> to perform the correction. You can also use the "Interactive Mode" if you wish. Consult Help for a discussion of the interactive mode.
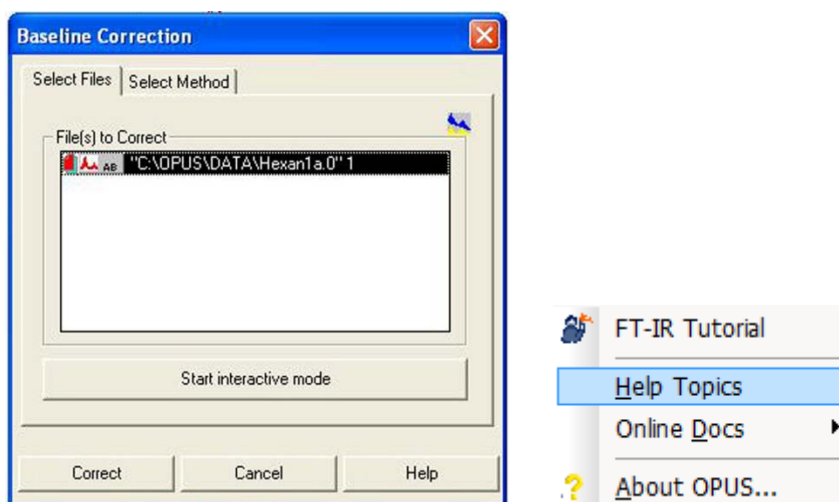


3. Peak Picking

Double-click on  to automatically pick and label significant picks in the displayed spectrum. To modify the parameters used, single-click on the icon to enter the Peak Picking view. Visit the Help page for a discussion of the interactive mode.

Start with *Sensitivity* set to a 5% threshold. To find more peaks, choose a lower number; to find fewer peaks, choose a higher number. The peak-picking spectral window can be selected in several ways on the "Frequency Range" tab. The "Y-limits" tab can be used to filter which peaks are labeled. Although there may be values set for these filters, we normally do not activate these filters. The settings on the Mode tab will normally be *"automatic peak detection", "standard method",* and *"overwrite the peak table"*. *"Append to peak table"* can be useful under some circumstances. Right-click in the Spectrum window and select *"Single Peak Pick"* to label peaks manually.
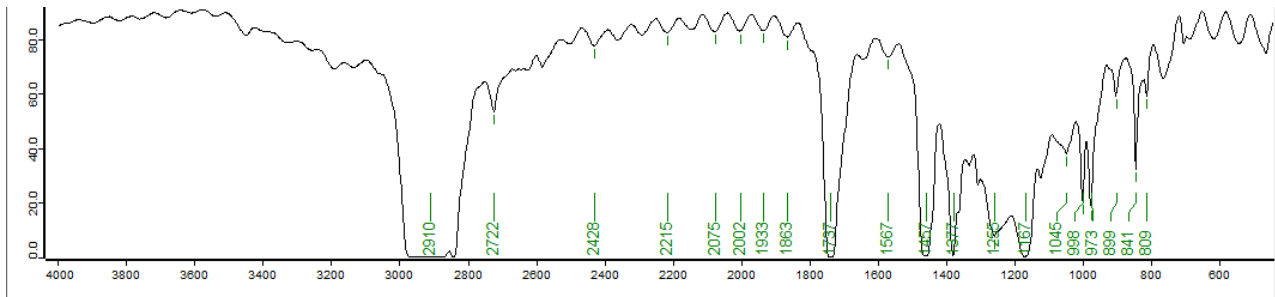
### 4. Spectral Subtraction

 Spectral subtraction is a special topic not covered in this basic guide, but it will be shown to you during your training. It is very useful. Review the OPUS help topics, and request assistance as necessary.

### 5. Printing the Spectrum



Use the Quick Print icon,  to print the spectrum displayed in the Spectral Window. Use File -> Print Setup… to select between the various printers that are available.

### 6. Saving Data

All data is instantly saved in the *C: \Opus_7.2.139.1294\Meas* directory with a filename *abc-mmddyy.xxx*, where **abc-** should be your assigned user initials. **Mmddyy** will be the date code, and **xxx** will be an incrementing number; both assigned automatically. If you modify the file (baseline correction, peak picking, et cetera) you will be prompted when you exit OPUS to save or discard the changes. Do NOT rename the file stored in the *C:\Opus_7.2.139.1294\Meas* directory!! This directory should only contain *the original files* named using the *abc-mmddyy.xxx* convention. If you wish to save modified files, or save files in a non-OPUS format for processing elsewhere, save them in your data space on the D-drive, e.g. *D:\Data\username*.

Use the "Save As" icon.  A dialog box will appear allowing you change the path to your space on the D-drive, change the name, and change the data format.