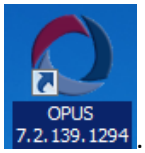
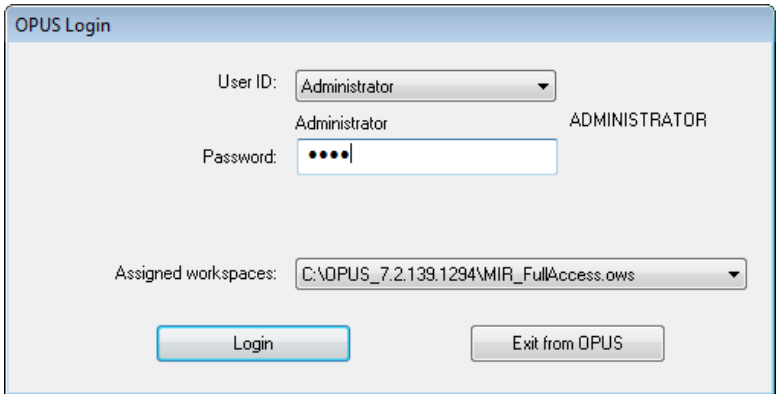
Using OPUS to Process Evolved Gas Data (8/12/15 edits highlighted)

Once FTIR data has been acquired for the gases evolved during your DSC/TGA run, you will process using the OPUS software package.

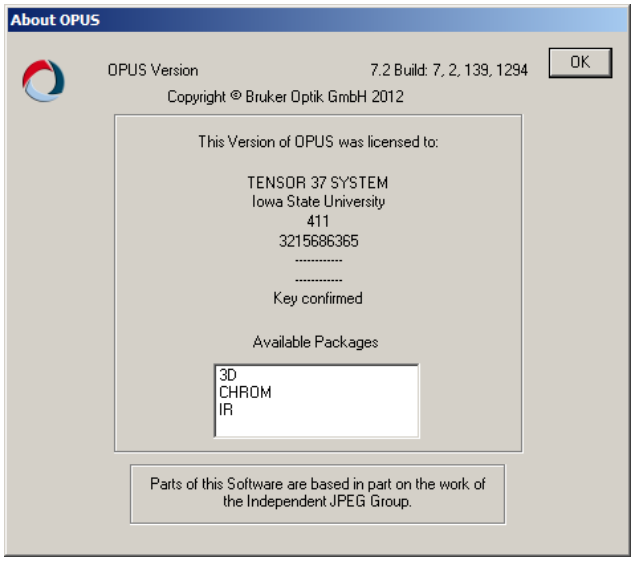
Select OPUS on your desktop 

.

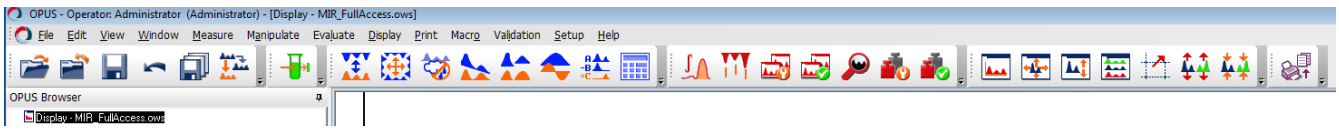
Login as Administrator; password= OPUS; Workspace= MIR_FullAccess.



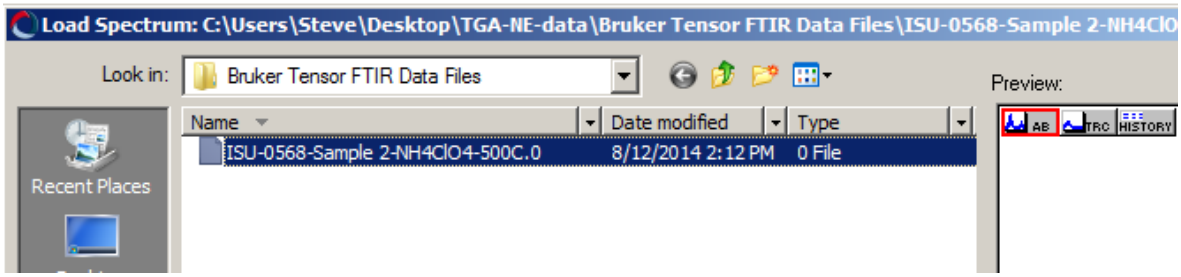
Say OK to the license manager prompt. Note that the 3D, CHROM, and IR packages must all be present.



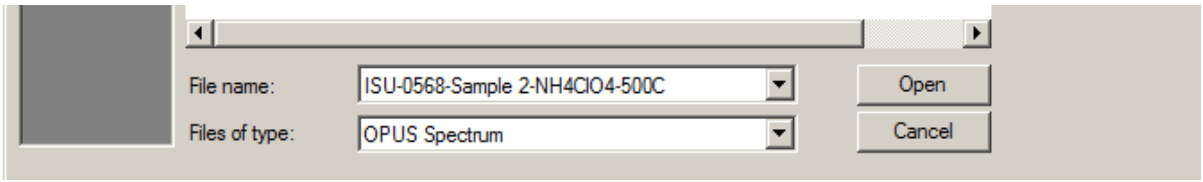
The OPUS view will look similar to the following, but it might not be identical, depending upon the workspace configurations.



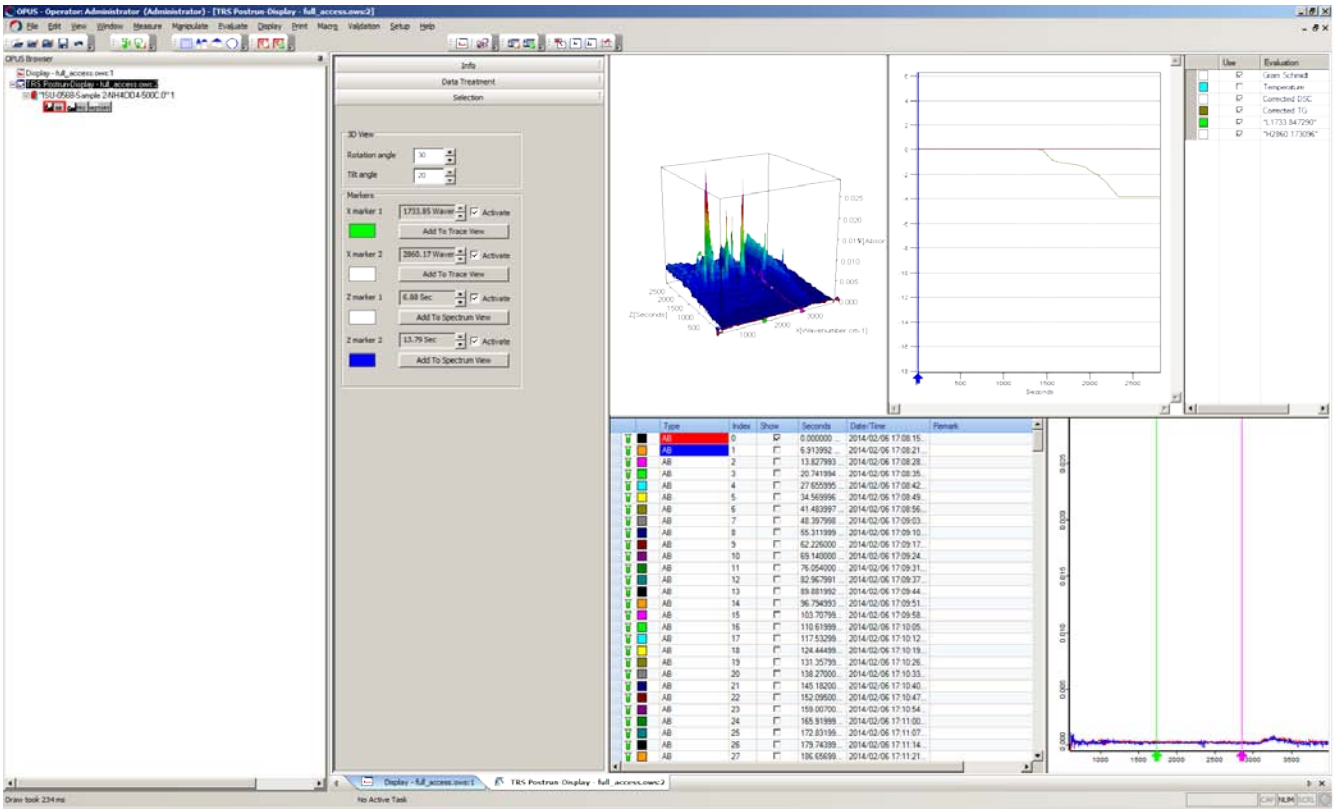
Using the File Load icon  , navigate to the folder with your FTIR data and select the file you wish to process. Note that the data blocks for the file must include a TRC (time resolved chromatography) data block.



Select and Open the file.



The TRS (time-resolved spectra) Postrun Display should open.

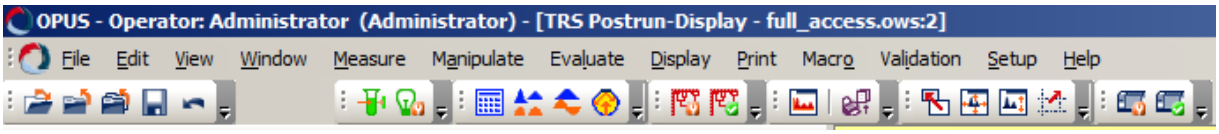


Let the data processing begin!!!

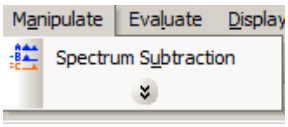
Parts of the OPUS Workspace

Let's briefly review the parts of the OPUS workspace view. The configuration of the view is very flexible. You might not see exactly what is shown in this tutorial, depending upon how the workspace has been set up.

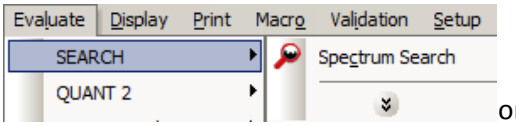
Task Bar and Pulldown Menus



Mouse over each icon to see a brief description of its function. You will be using icons in the zoom  and file management groups  quite frequently. You will be using Spectral Subtraction from the Manipulate pull-down and Search Spectrum from the Evaluate pull-down.

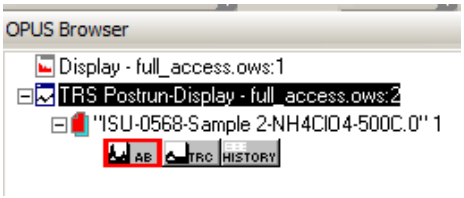


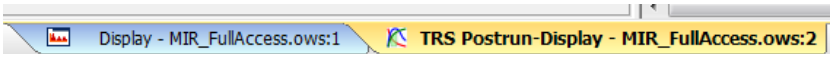
or 



or 

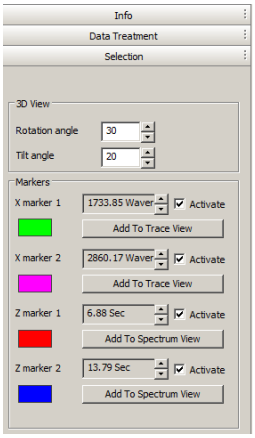
OPUS Browser

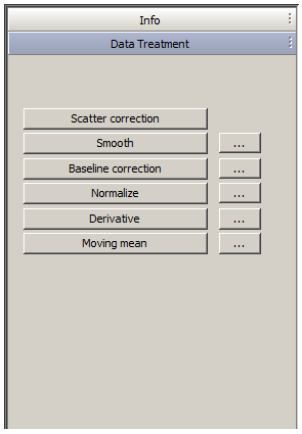


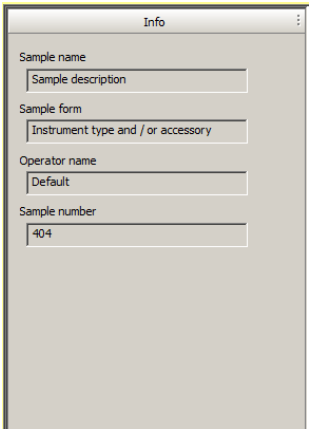


Initially, only the 3-D file you opened will be in the browser. Note the TRC data block. This triggers the workspace view to open a TRS Postrun display tab as well as the normal Display tab. Until we extract spectra from the 3-D view, there will be no Display entries (spectra) in the browser

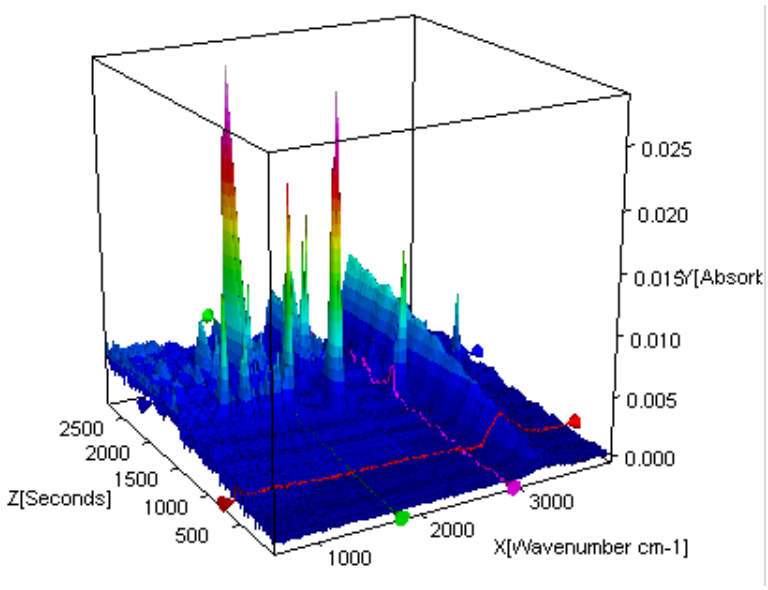
Info / Data Treatment / Selection (Not critical to this workflow)







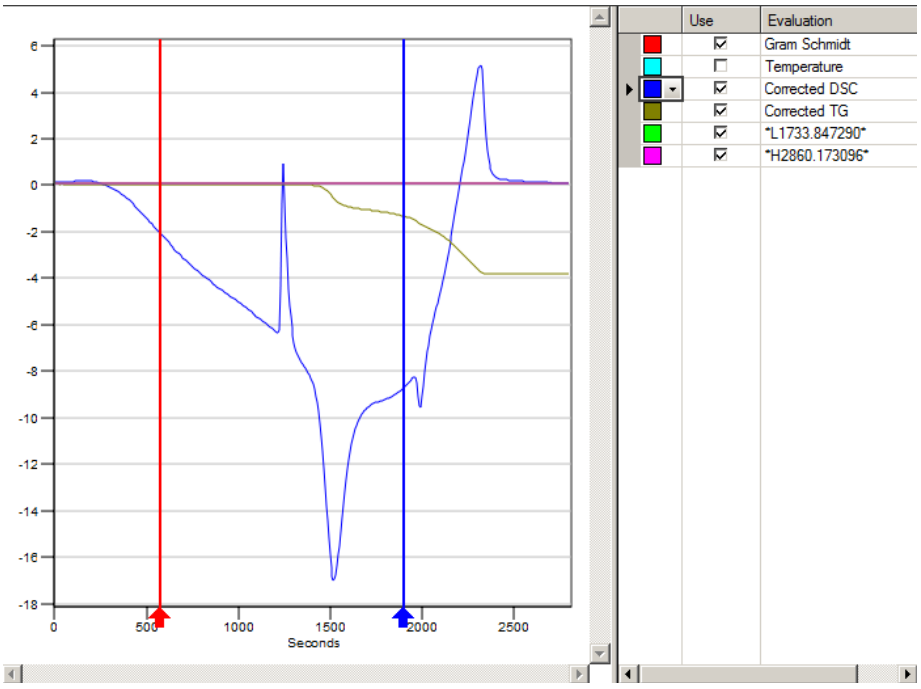
Three-D Plot



Note the red and blue handles on the Z (seconds) access, and the green and pink handles on the X (wavenumber) axis. The Y-axis is amplitude (absorbance units). You will also see the red/blue handles in the Z-Y curves view, and you will see the green/pink handles in the X-Y curves view. You can grab the handles and move them in any view and the changes will also be seen in the other views.

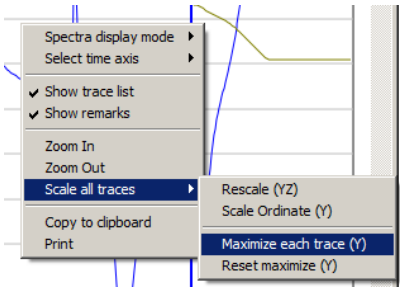
Z-Y Curves

A range of 2-D curves can be viewed by selecting from the list on the right side of the view.

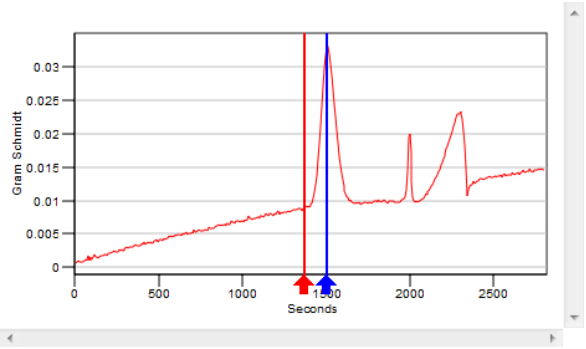


Note that the color of each trace can be changed by left-clicking next to the existing color square, left-clicking on the resultant down-arrow, then left-clicking on a different color in the color palette presented.

Mouse-over a point in the curve to see the corresponding time. Use the right mouse button to active a range of zoom and scale functions for the display. "Maximize each trace" is sometimes useful. Deselecting traces you don't need at the moment is also useful.



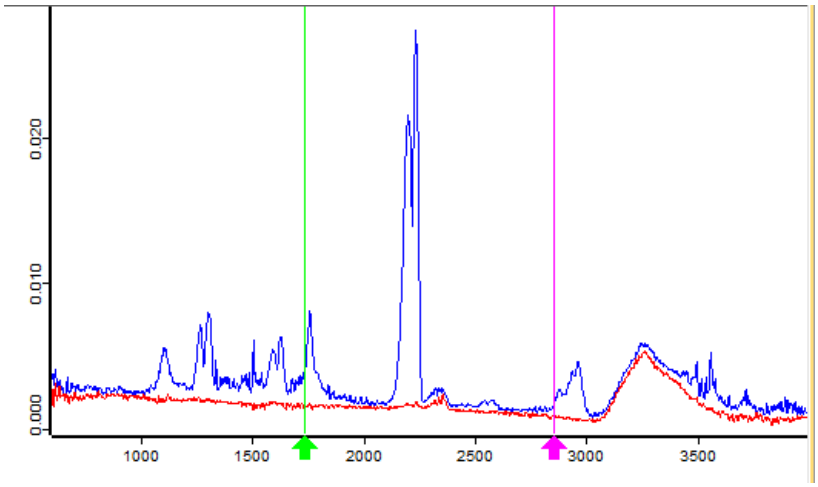
If we choose to view just the Gram-Schmidt (trace maximized in the ordinate), we should see:



Drag the blue cursor to the top of the first G-S peak, and drag the red cursor to the baseline just before the peak begins.

X-Y Curves (Spectra)

You should now see three spectra in the X-Y view, corresponding to the unknown peak selected by the blue cursor, the baseline scan selected by the red cursor, and a scan in black that corresponds to the Time= zero scan.

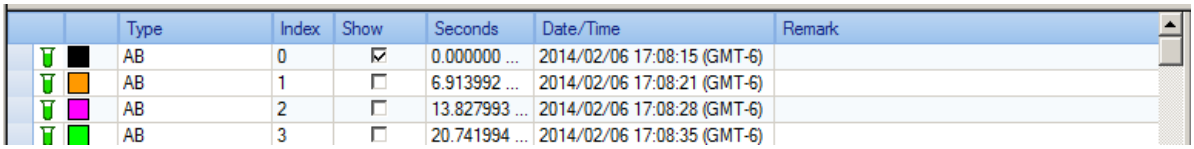


Explore the various functions available with a right-click of the mouse. The functions will vary depending upon whether you right-click in white space or right-click on one of the traces. This is common behavior in most of the views.

Note that it is NOT possible to right-click and “remove from display”. That feature pertains to “Display” views, not “TRS Postrun Display” views. Instead, you will deselect in the Scan List to remove from the view.

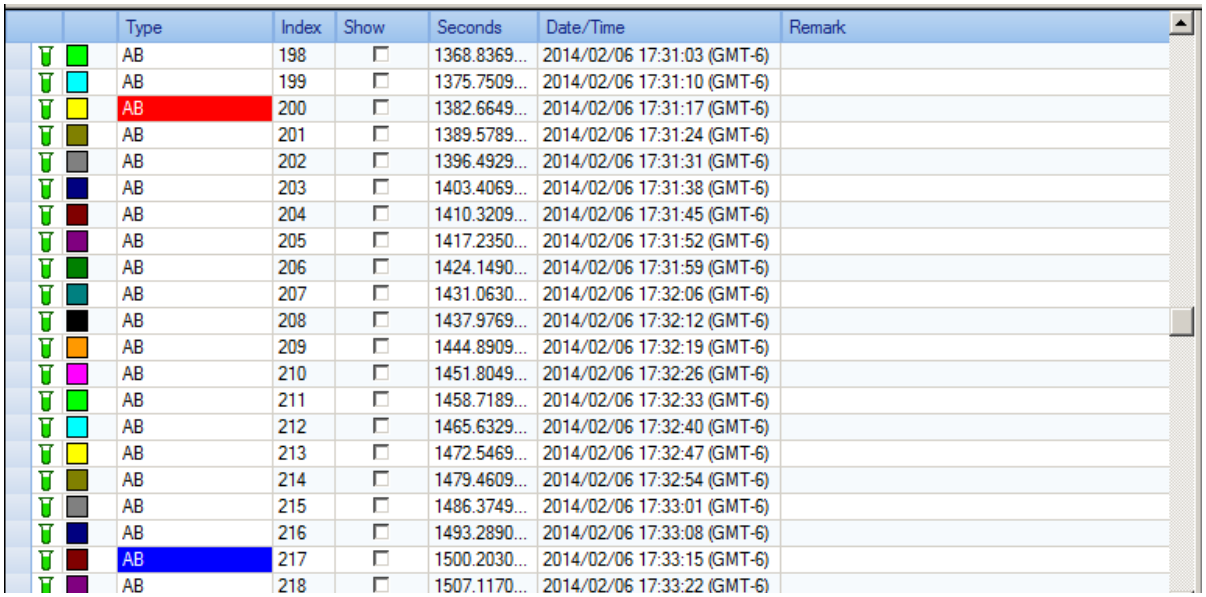
Scan List

The tga.xpm parameter set results in approximately four complete spectra per minute. The Scan List provides time and index info about each spectrum and also reflects which scans have been selected for viewing. Note that the time=0 scan is selected for viewing by default (the black curve). Uncheck the “Show” box to remove it from the display.



Type	Index	Show	Seconds	Date/Time	Remark
AB	0	☑	0.000000 ...	2014/02/06 17:08:15 (GMT-6)	
AB	1	☐	6.913992 ...	2014/02/06 17:08:21 (GMT-6)	
AB	2	☐	13.827993 ...	2014/02/06 17:08:28 (GMT-6)	
AB	3	☐	20.741994 ...	2014/02/06 17:08:35 (GMT-6)	

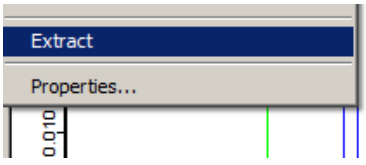
Scrolling down will eventually show you the red/blue scans currently selected for display.

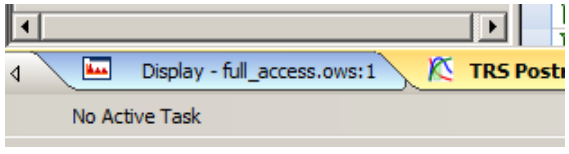


Type	Index	Show	Seconds	Date/Time	Remark
AB	198	☐	1368.8369...	2014/02/06 17:31:03 (GMT-6)	
AB	199	☐	1375.7509...	2014/02/06 17:31:10 (GMT-6)	
AB	200	☐	1382.6649...	2014/02/06 17:31:17 (GMT-6)	
AB	201	☐	1389.5789...	2014/02/06 17:31:24 (GMT-6)	
AB	202	☐	1396.4929...	2014/02/06 17:31:31 (GMT-6)	
AB	203	☐	1403.4069...	2014/02/06 17:31:38 (GMT-6)	
AB	204	☐	1410.3209...	2014/02/06 17:31:45 (GMT-6)	
AB	205	☐	1417.2350...	2014/02/06 17:31:52 (GMT-6)	
AB	206	☐	1424.1490...	2014/02/06 17:31:59 (GMT-6)	
AB	207	☐	1431.0630...	2014/02/06 17:32:06 (GMT-6)	
AB	208	☐	1437.9769...	2014/02/06 17:32:12 (GMT-6)	
AB	209	☐	1444.8909...	2014/02/06 17:32:19 (GMT-6)	
AB	210	☐	1451.8049...	2014/02/06 17:32:26 (GMT-6)	
AB	211	☐	1458.7189...	2014/02/06 17:32:33 (GMT-6)	
AB	212	☐	1465.6329...	2014/02/06 17:32:40 (GMT-6)	
AB	213	☐	1472.5469...	2014/02/06 17:32:47 (GMT-6)	
AB	214	☐	1479.4609...	2014/02/06 17:32:54 (GMT-6)	
AB	215	☐	1486.3749...	2014/02/06 17:33:01 (GMT-6)	
AB	216	☐	1493.2890...	2014/02/06 17:33:08 (GMT-6)	
AB	217	☐	1500.2030...	2014/02/06 17:33:15 (GMT-6)	
AB	218	☐	1507.1170...	2014/02/06 17:33:22 (GMT-6)	

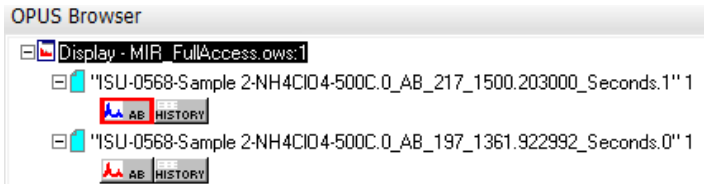
Extracting Spectra for Processing

In the spectra view, right-click and extract the blue spectrum corresponding to the first peak in the G-S trace. Then right-click and extract the red spectrum corresponding to the baseline just before the first peak in the G-S trace. Then select the Display tab.

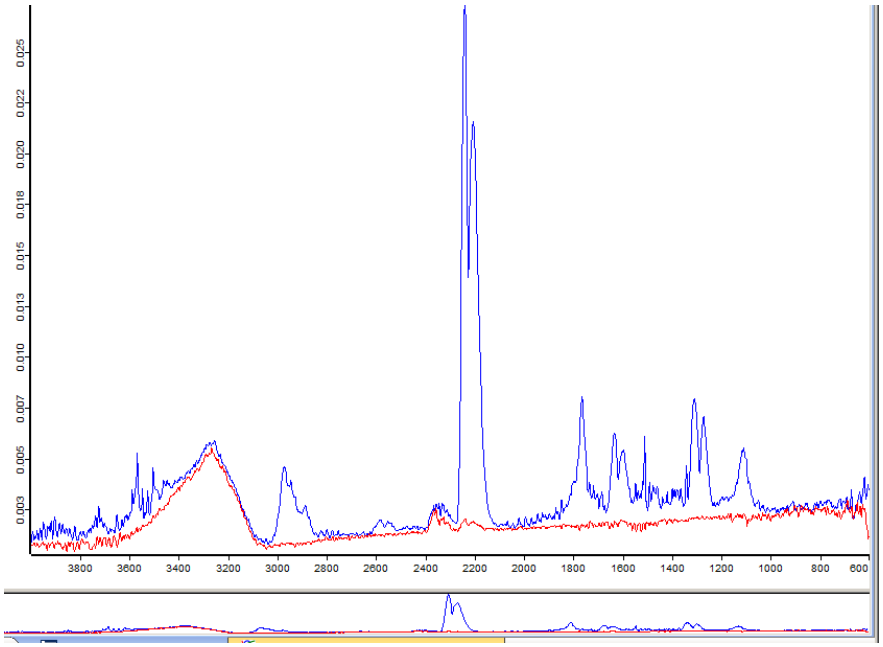




Note that the OPUS browser now lists two unmodified (no red cover on the folder icon) spectra as “Display” files, and still lists the modified (red cover flag) 3-D file as a “TRS Postrun Display” file. The extracted spectra files have been automatically named to include the index number and time stamp.

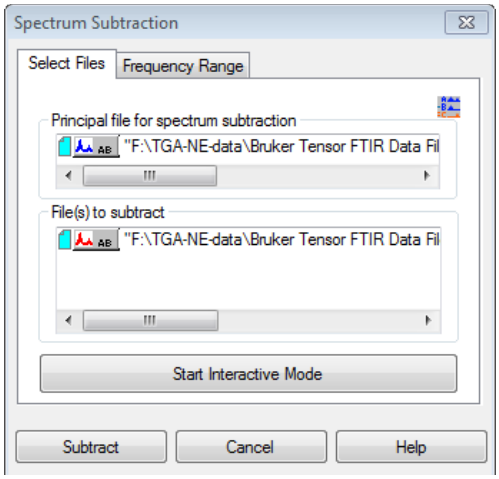


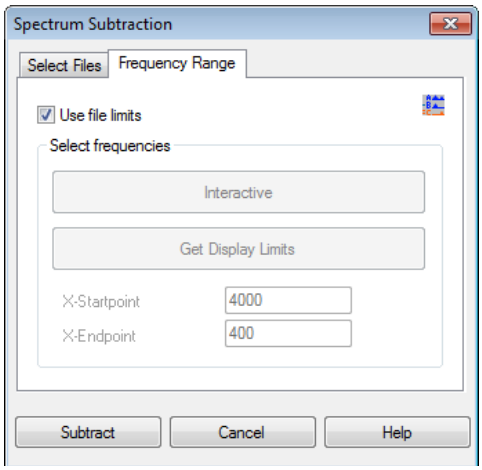
In the spectrum view, the spectra are probably not normalized in the Y-axis. Use the Scale Ordinate button  to resize. The view should look like this:



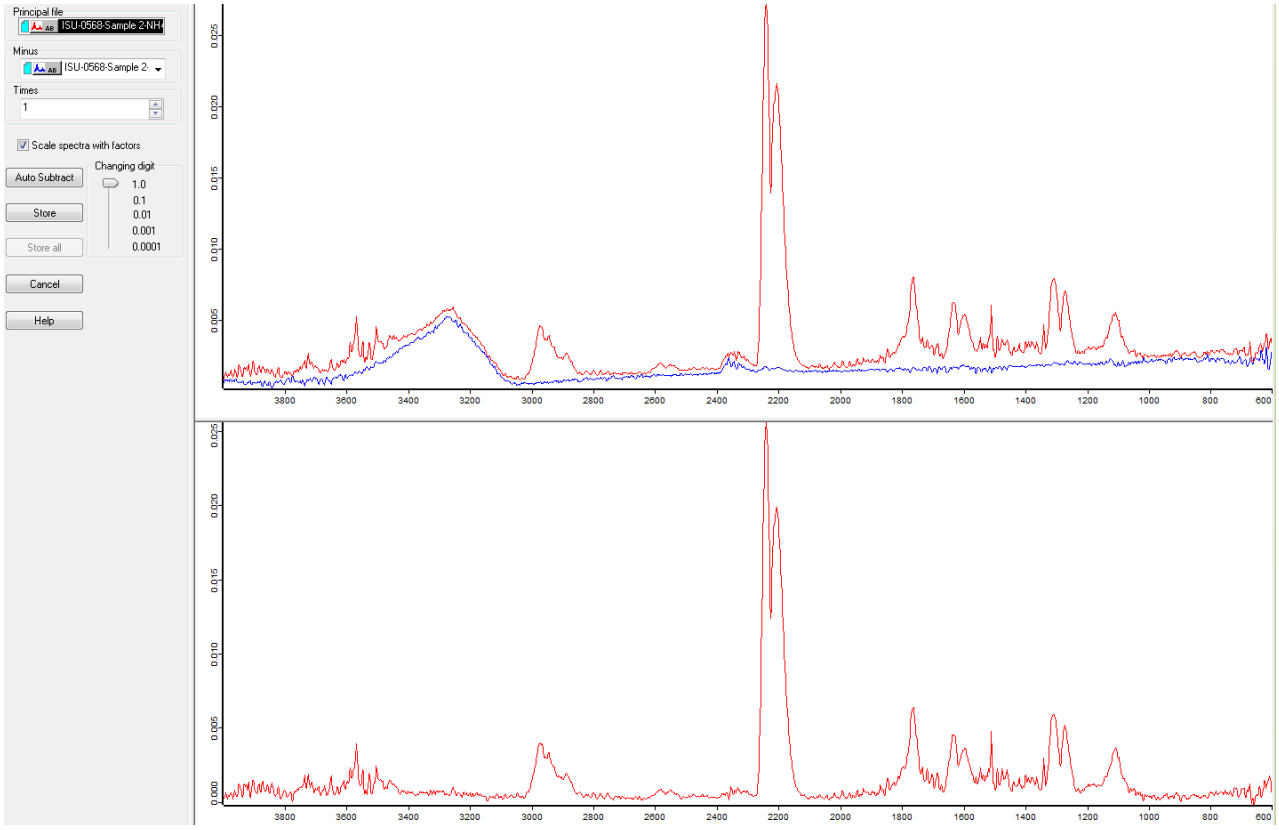
Spectral Subtraction

Make sure that the AB block for scan 217 (unknown peak) is selected in the browser. Open the Subtraction view  . Verify that the correct file is selected as the Principal file for spectral subtraction. In the browser, left-click and drag the AB data block for scan 197 (baseline) into the File(s) to be subtracted box. Verify that the frequency range for subtraction is set to “Use File Limits”. The views should look like:





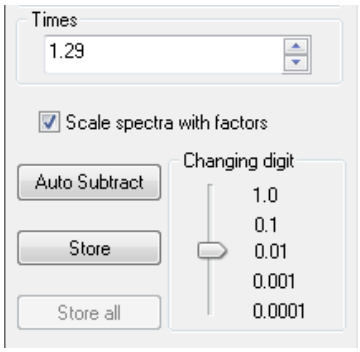
Select "Start Interactive Mode". The view should look like:



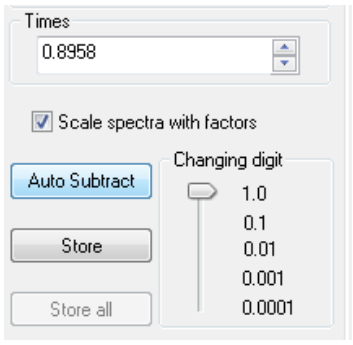
Note that the colors of the files have swapped! The Principal is now shown in red, and the file to be subtracted is now shown in Blue. This is not easily changed, so please be aware of it. The Principal and subtraction files are shown superimposed in the top trace. The resultant spectrum (after subtraction) is shown in the lower trace. Note that the initial subtraction constant used is Times = 1. Change this multiplication factor and observe the result. Note that the blue spectrum scales as you do this.

Change the Digit value from 1.0 to 0.1. The multiplication factor can now be incremented in smaller steps. In some cases, it might be useful to change the Digit value to 0.01 in order to achieve the optimum subtraction constant. Your selection of a subtraction constant will depend upon which region of the spectrum is most important to you! There is no single value that is optimum for ALL peaks in the spectrum. Alternatively, select

"Auto Subtract" and the software will try to decide for you. You might also want to change the frequency range for subtraction by selecting "cancel" and making a different choice on the Frequency Range Tab.

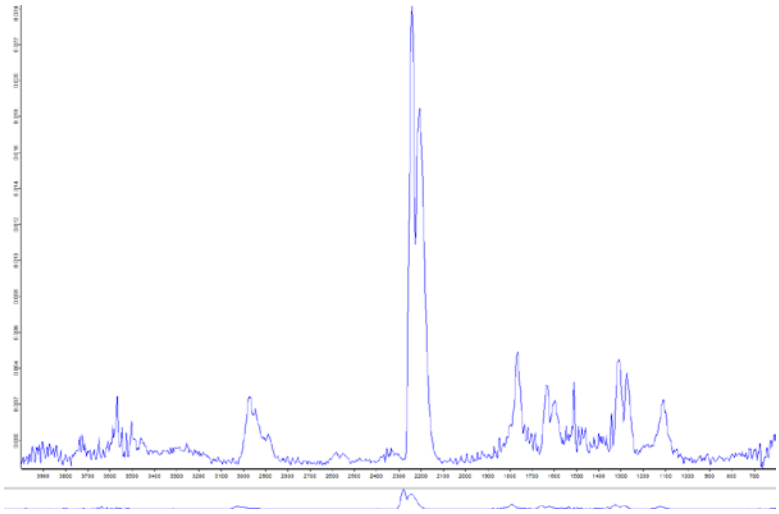


Manual setting



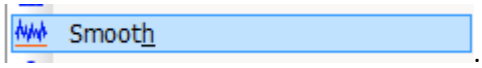
Result of "Auto Subtract"

Select "Store" when you are satisfied. The subtraction program will close and a new data block (subtraction info) will be added in the browser line for scan 217. Note the red flag on the folder cover indicating that the spectrum has been modified. The modified spectrum is shown in the Display view. Remove scan 197 from the display if it is still present. The view should now look like:

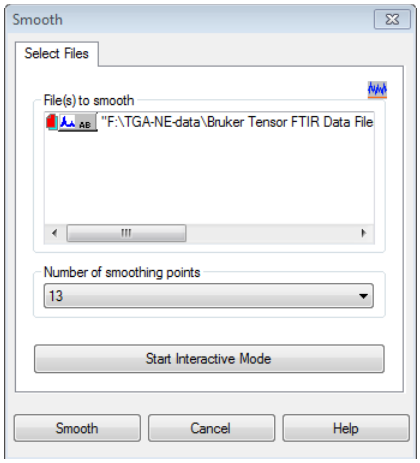


Smoothing Spectra

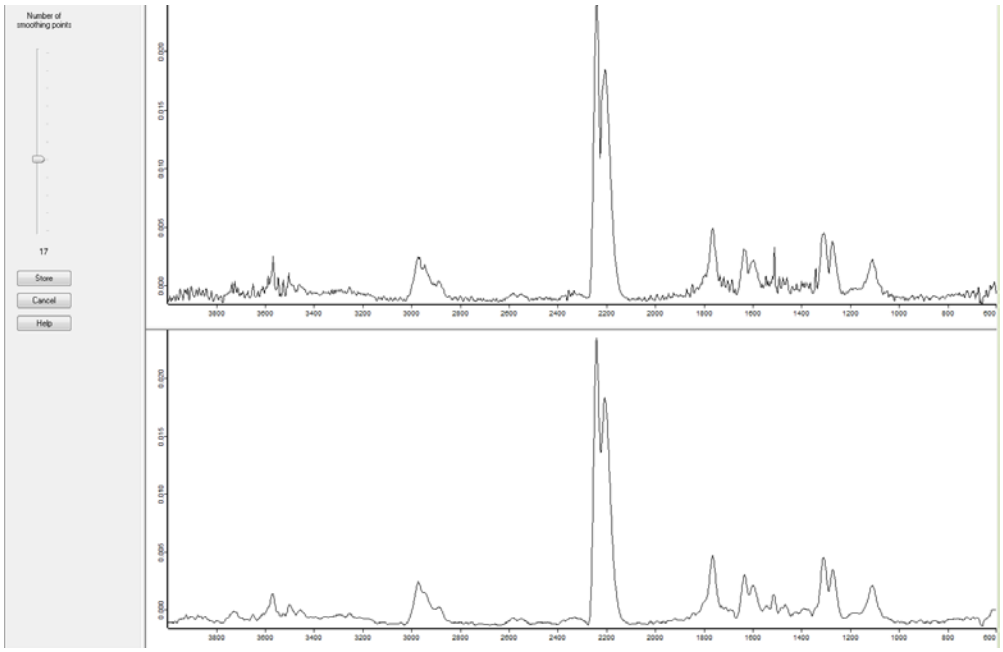
Make sure the correct data block is selected (red box) in the OPUS browser. Select the Smoothing icon if it is on the ribbon bar, or select it from the Manipulate pull-down.



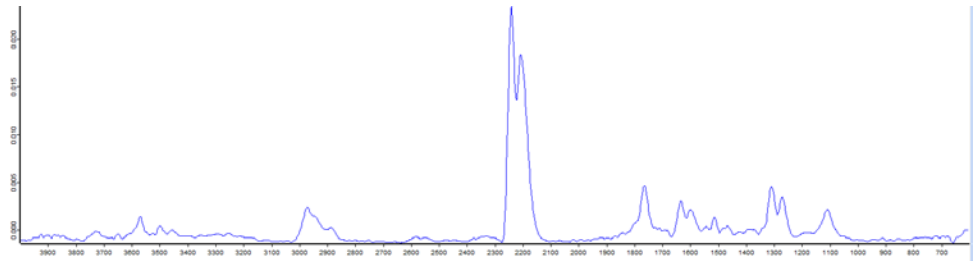
You should see:



The number of smoothing points used can range from 5 (minimal smoothing) to 25 (maximum smoothing). Set a reasonable value and then select “Smooth”, or select “Start Interactive Mode” if you are unsure which smoothing value to use. Try to find the value that minimizes “noise” while preserving the spectral features of interest to you. Both the unsmoothed and smoothed spectra are shown.

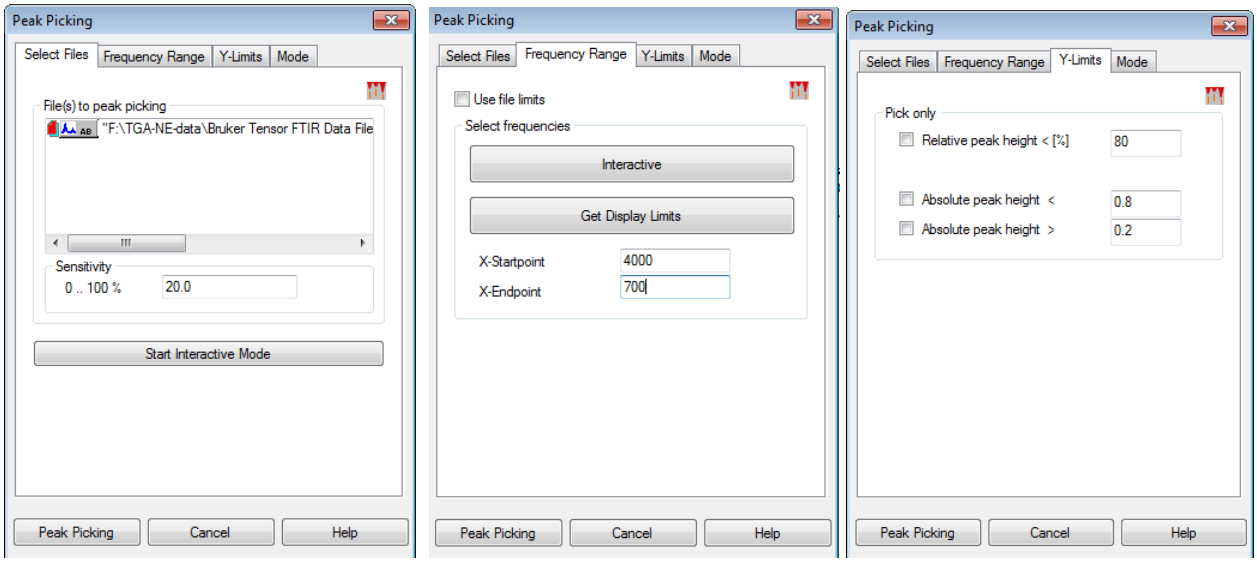


Select “Store” to end the Smooth program. The modified spectrum is shown in the Display view.



Peak Labelling

Make sure the correct data block is selected in the OPUS browser. Select Peak Picking from the ribbon bar or from the Evaluate pull-down.

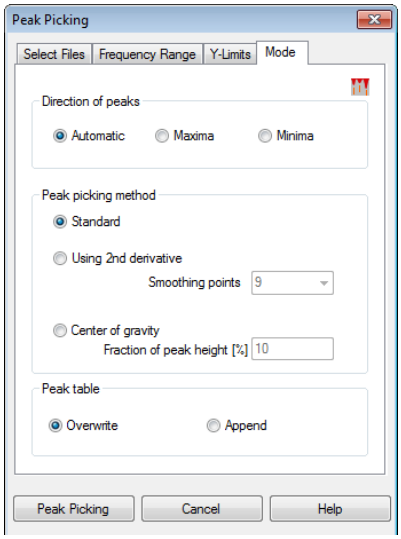


Note that the Sensitivity selection is in %, and represents a horizontal threshold above which peaks will be added to the peak table. Therefore lower numbers find more peaks, higher numbers find fewer peaks. I find the “Start Interactive Mode” selection to be less useful in this program.

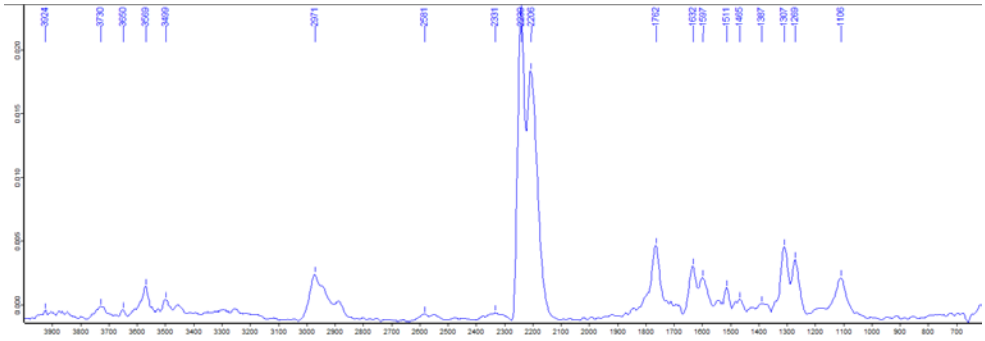
Note the various selections available on the “Frequency Range” tab. Only peaks within the selected frequency list will be added to the peak table.

The “Y-Limits” tab allows various filters for selecting peaks from the peak table for labelling on the display.

On the “Mode”tab, the normal selections should be *automatic, standard*, and *overwrite*.

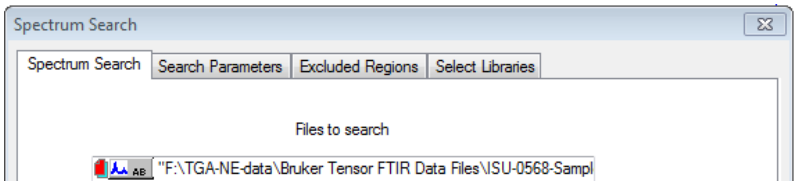


A Sensitivity selection of 2% should give the following result:

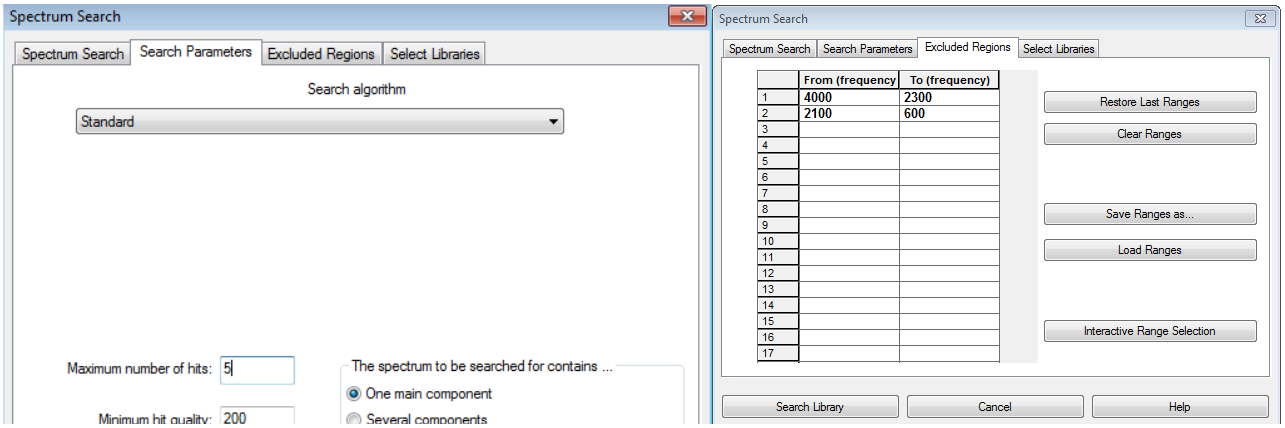


Spectrum Search

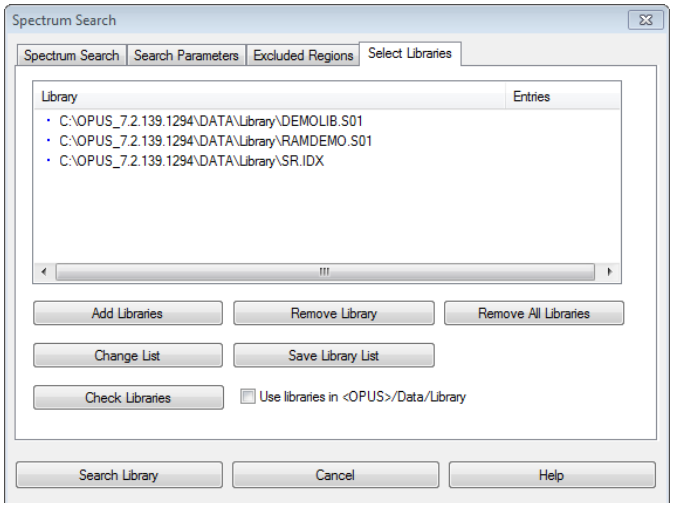
Make sure the correct data block is selected in the OPUS browser. Select Spectrum Search  from the ribbon bar or from the Evaluate pull-down.

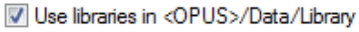


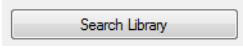
On the Search Parameters tab the Minimum hit quality and other parameters can be optimized. For this example, set Maximum number of hits to 5, hit quality to 200 (a perfect match is 1000), and check the “One main component” radio button. The “Excluded Regions” tab can be used to define regions that will not be considered in the match calculation. In this example, exclude all of the peaks above 2300 and below 2100 wavenumbers in order to force the search to help identify the structural features in the region between 2100-2300 wavenumbers.



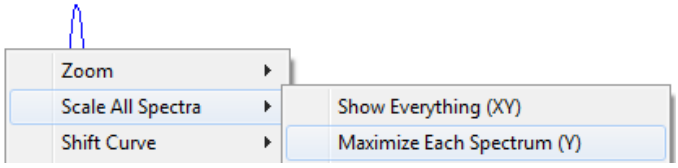
Note on the "Select Libraries" tab that the program will search by default all libraries in the <OPUS>/Data/Library path. Unchecking the default selection allows you to be more selective if you have imported targeted libraries or created libraries of your own. [We now have the NIST vapor phase library!]



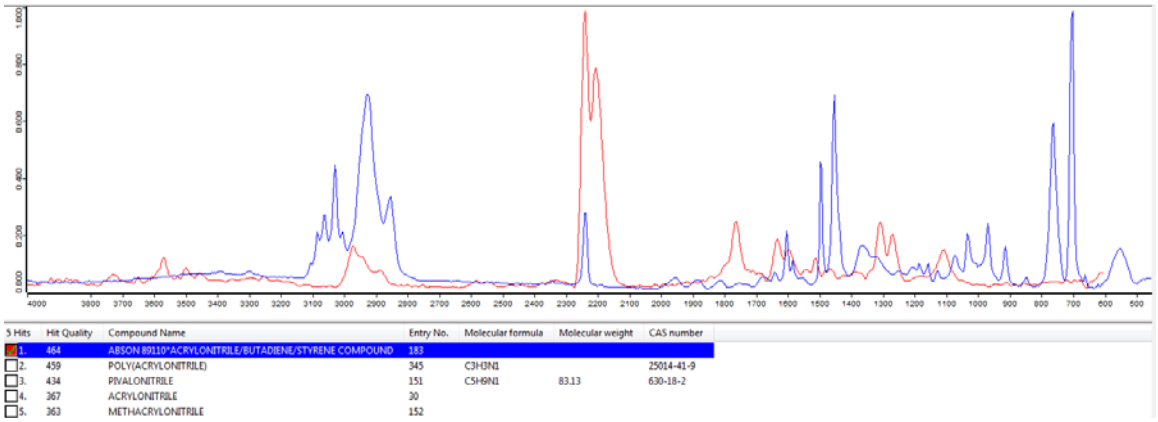
Go back to 

Selecting Search Library 

hould then give a list of the five best matches (considering only the narrow spectral region chosen) and the view will default to your spectrum and one result from the list of 5. At this point it may be necessary to right-click in the spectral display and choose "Scale All Spectra -> Maximize Each Spectrum in the Y-axis."



You should now see:



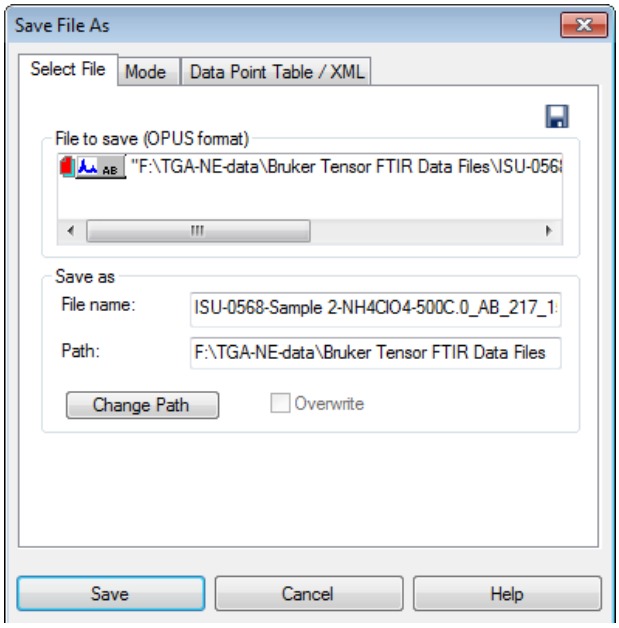
If you select from the other hits in the list, you will see that NONE of the five best matches have the characteristic peak doublet seen in the unknown, even after we restricted the library search to consider only that spectral region in choosing best matches.

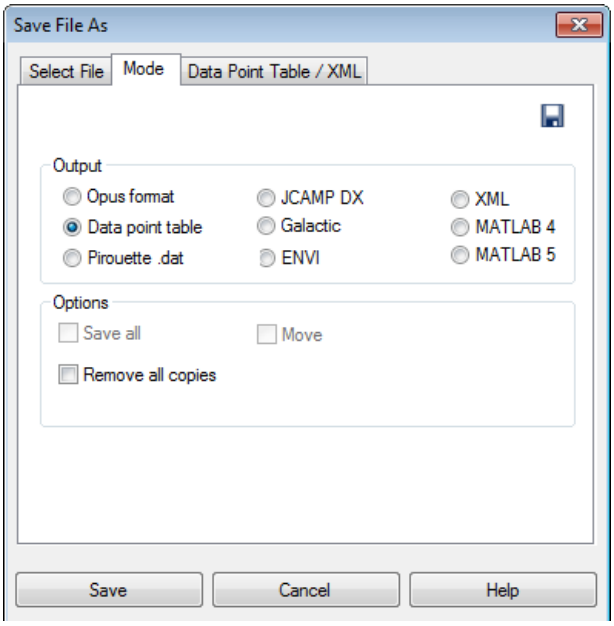
At this point you might want to redo the search, allowing for more hits perhaps, and visually review all of those. The library included with our OPUS purchase only has 355 compounds. It is certainly possible that the unknown is not among them. [We now have the NIST vapor phase library!]

OPUS does allow you to create user libraries, or add new entries into the library that was delivered with the instrument.

Printing, Saving and Exporting Spectra

As mentioned earlier, the spectra you have extracted from the original 3-D data file are saved automatically in the same location with embedded index number and time stamps in the extended filename. You may also choose to select any spectrum (data block) and export it using the "Save File As"  option.

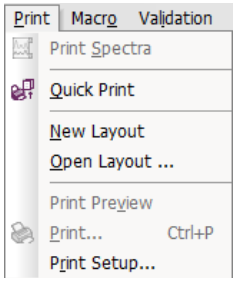




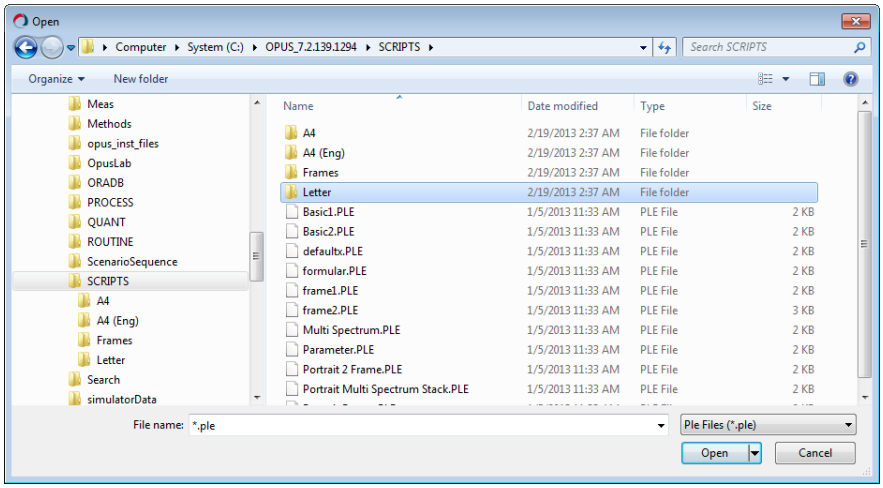
Note that the path or filename can be changed. The Mode tab allows selection from a variety of IR data formats commonly in use. The simplest selection is “Data Point Table” which creates text-readable X-Y pairs.

Printing is not possible from the File pull-down (although Print Setup is active).

The “Quick Print” icon  would routinely be used to print spectra, based upon settings managed from the Print pull-down. Be sure that “letter” size paper (the default seems to be A4) and “landscape” mode are selected in “Print Setup…”.



There are HUNDREDS of report layouts to choose from, all of which can be customized and saved.



Opening a report layout will show the graphic and text elements and allow resizing or relocation of the elements or the inclusion of additional elements.