

CH 362

Use of TopSpin v. 3.5 for 1-D NMR Spectrum Processing and Printing

General Instructions

When the TA runs your spectrum, it will generate a folder of data with about a dozen files (and some subdirectories). You will need to save this folder somewhere that you can get to it. The best is to save it to the T: drive for access from the GbAd 409 computers. Several other options:

- Have the TA zip the folder, then email it to yourself.
- Save the folder to your GoogleDrive space, Box, Dropbox or other cloud space

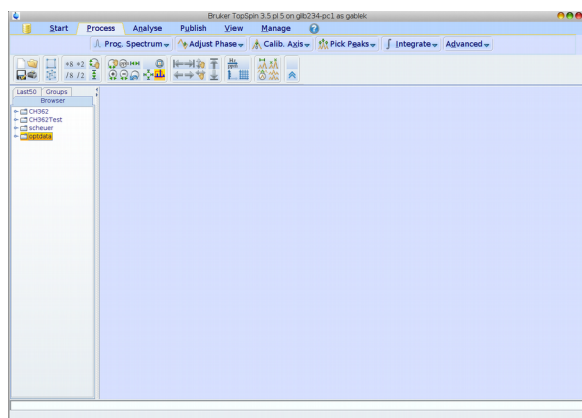
Note that due to security issues, it is not possible to mount a thumb drive to the computer in GbAd 101. TopSpin is available in the six laptops in GbAd 409, and we can help you install it on your request on your computer. The license allows noncommercial (academic) use like you will be doing.

In all likelihood, the TA will have stepped you through the basics of doing a Fourier Transform and providing basic phase correction. This tutorial will have you start from the FID, though you may choose to use the processed data and start from Step 8 below (though you might also need some phase adjustment, Step 6).

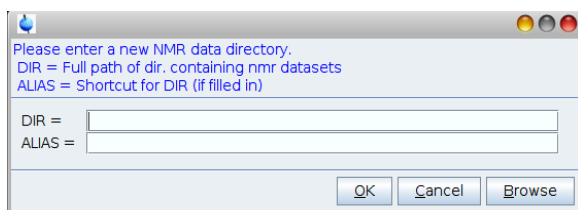
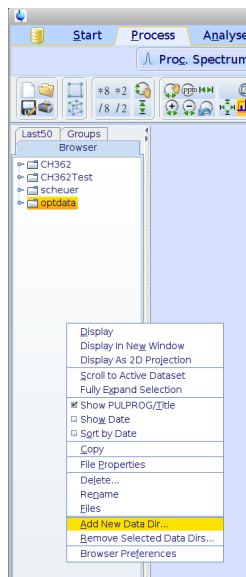
1. Place the data folder on the computer you are using. If you are using the GbAd 409 computers, there is a shared folder available at T:\ChemLabs\chem362\Winter2017\NMRData; you may have used this area for IR data earlier. Be certain your folder has a unique name (ideally your name and notebook page number).

2. Open TopSpin.

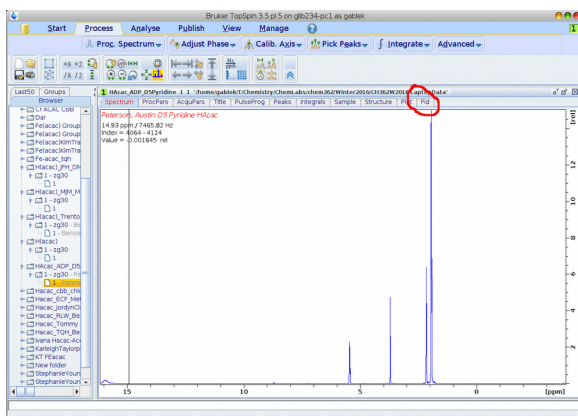
You will get the following screen.



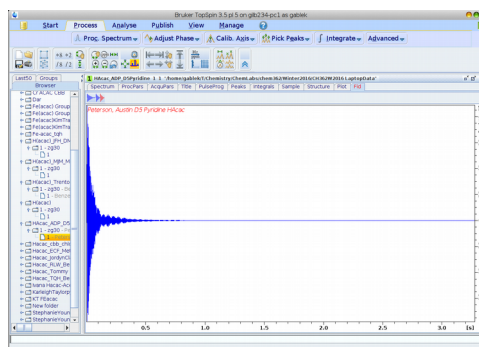
If you are using your own computer (but NOT in GbAd 409!), you now have to add the folder where you saved your data to the list of searchable data on the left. Left-click, and select “Add New Data Dir”. Select “Browse” and find your data folder. Hit “OK.”



3. Now open your data. You will likely have to locate a subheading titled “zg30” after the experiment that you ran to collect the 1D NMR data. Under that is the process number—double click on that. Now things will look like this:



As noted, any processing you did on the spectrometer is there. To reprocess, select the FID (red circle). Now you see:

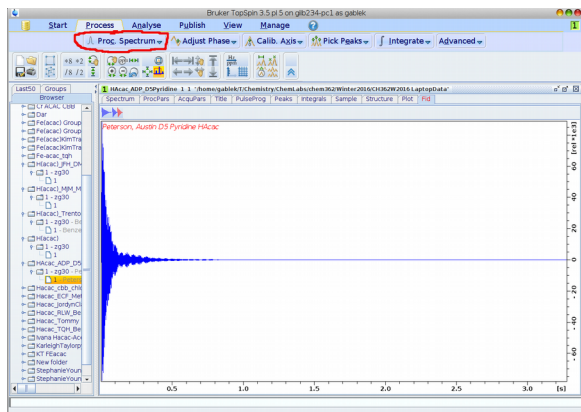


4. Your goal is to get a processed spectrum that identifies all the peaks by chemical shift and reports the integrals. To get this, the order of events is:

1. Apply a “window function” so that the peaks are well-behaved.
2. Apply phase correction to make certain each peak starts and ends on the baseline.
3. If necessary, correct the baseline for any curvature.
4. If necessary, set the reference (solvent line) chemical shift correctly.
5. Pick peaks.
6. Integrate.
7. Print/save to a PDF.

The two menu bars across the top are how you navigate to each.

5. Window function and fourier transform: use “Proc. Spectrum.”



Configure Standard Processing (proc1d)

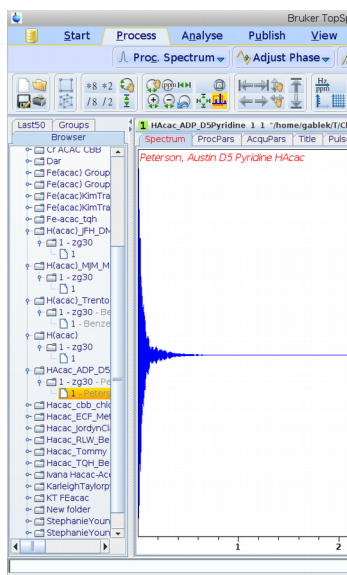
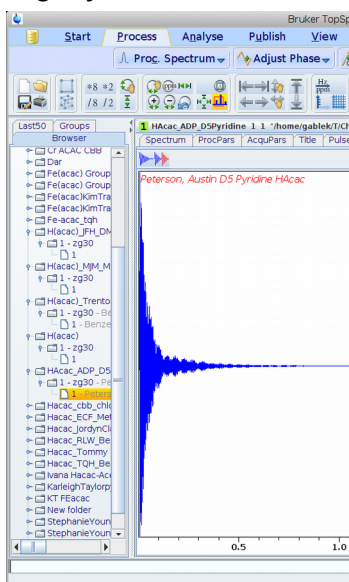
Window Multiplication (wm)

Fourier Transform (ft)

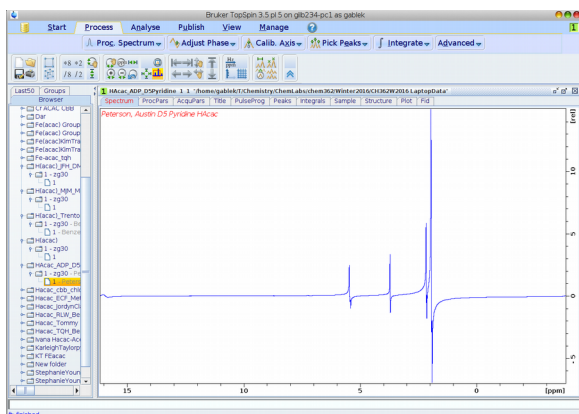
Fourier Transform Options ... (ftf)

Start Automation AU Program (xaup)

The defaults should be fine, so hit “OK” at the next popup. The shape of the FID will change slightly:



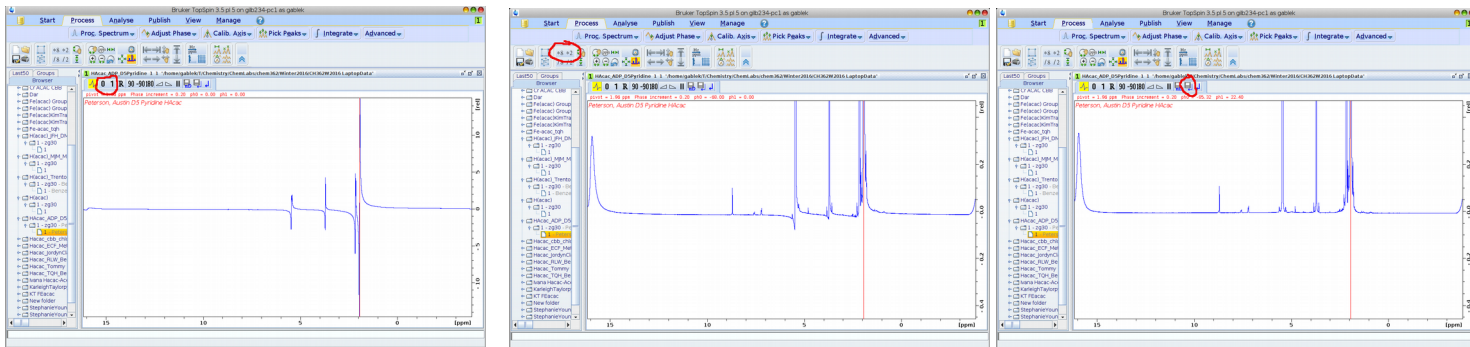
Now “Fourier Transform” under the “Proc. Spectrum” menu does just that:



Note that the peaks are not even with the baseline; this reflects the need to adjust the “phase” of the spectrum. (This derives from where during the set of sine waves in the signal we choose to start data acquisition.)

6. Phase adjustment. Clicking “Adjust phase” enters the manual adjustment routine. (Automatic phase adjustment usually works well, but some manual adjustment is really necessary to get good integration.)

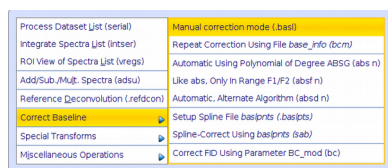
You will click and drag (up/down) on the “0” and “1” (circled). Each has its largest impact on one side of the spectrum. Start with “0” first, then go to “1”. You'll probably have to go back and forth several times. Also, be sure to do a vertical expansion (“*2” or “*8” on the icon bar).



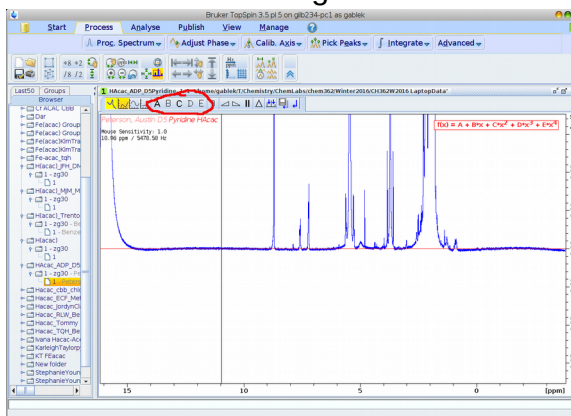
When all the peaks are even with the baseline save and return out of the phase routine (icon bar above the spectrum, next-to-the-right icon). If you get lost, the rightmost icon will undo all of your phase changes and you can start from scratch.

7. Referencing: This should not be necessary if you and the TA set up data collection using the correct solvent. However, you can adjust as necessary by clicking on “Calib. Axis”, left-clicking on the peak to be referenced, and entering the chemical shift.

8. Baseline correction. Expand the vertical scale (*8 one or more times) to see if there is any curvature in the baseline. Then select “Advanced” and “Correct Baseline” then “Manual Correction Mode”.

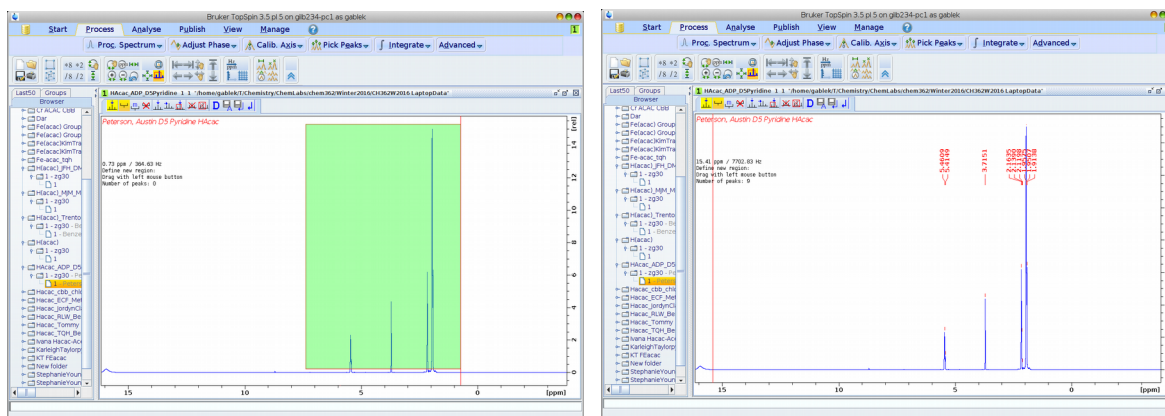


You will see the following screen:



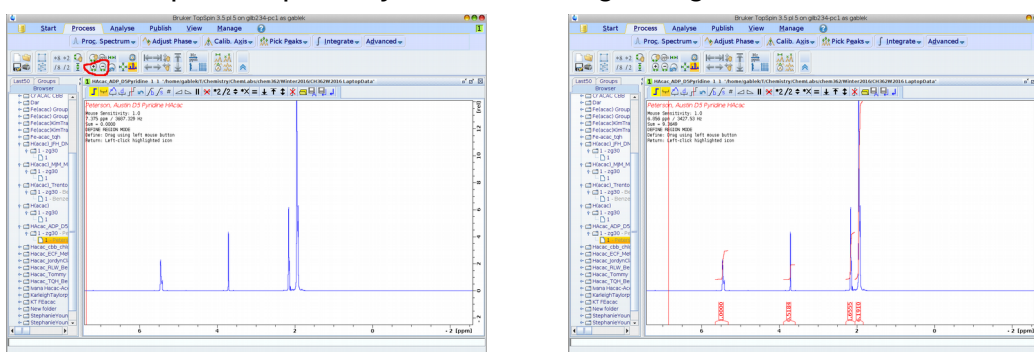
Adjust the baseline using the A, B, C, D, E controls and sliding up/down (as you did with phase correction). When the red polynomial matches the curve of the baseline, save & return. This particular spectrum does not need significant baseline correction, but yours might.

9. Peak picking. Select “Pick Peaks” and then click-and-drag over the peaks in the spectrum. Be careful: you do not want to get too close to the baseline or you'll be selecting noise (or things like ^{13}C satellites). The peaks will appear as vertical numbers with a line:



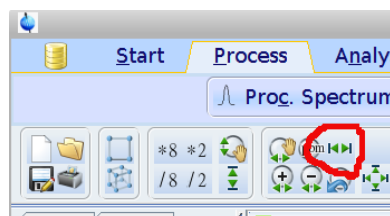
Save & return as always.

10. Integrate. Select “Integrate”, then click and drag across each peak. (It's best to do a horizontal expansion first to make sure you can cut the integrals closely). Then click-and-drag across each peak separately to cut the integral regions.

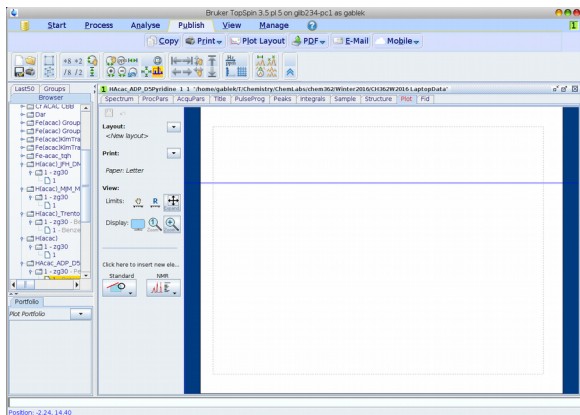


The integrals appear as numbers under each peak, with the cut region specified; the red vertical curves are proportional to the curve integral. To normalize (if you know the number of protons present in a particular peak), right-click on the peak, select “Calibrate Current Integral” and enter the number of protons you think are responsible for the peak. For Hacac this is best done as either “1” for the methine CH in the enol form, or “2” for the methylene CH_2 peak in the keto form.

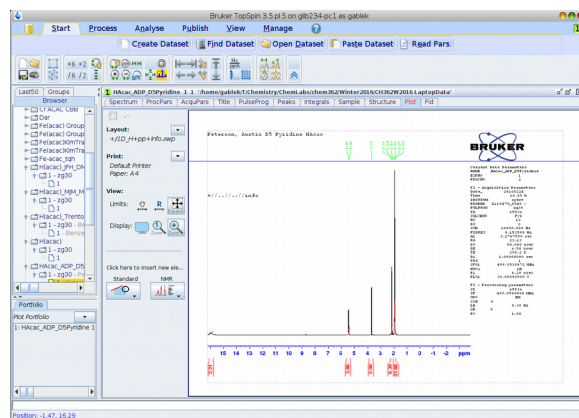
Undo the zoom:



10. Printing and saving. Select “Publish” and “Plot Layout.” You'll see this:



Under “Layout”, click the arrow and select “Open”, then “1D_H+pp+info.xwp”. The spectrum will now be displayed:



Change the default page size to “Letter” using “Print”, “Page Setup” and “Size.”

At this point you can print directly if connected to a printer, or you can save a PDF (“PDF” on the menu bar).