

Lab 3
Processing 2D NMR Data Offline
Report Due: Wednesday, November 5

Introduction

Just as you downloaded 1D FID data and worked that data up on your on computer in Lab 1, in Lab 3 you will do the same thing for 2D data. The advantages of working up your 2D data from your own computer are also the same; you can print whatever regions of the spectrum you like, and manipulate the data how you like, from the comfort of your desk. Maybe just as important, you can focus in on crosspeaks, adjust vertical range, and look more closely at features you might not have caught when you first acquired and analyzed the data, right on your computer.

The purpose of this lab is to have you download, process and print a 2D NMR spectrum from a remote computer. To do this, you will learn how to:

1. Use an NMR processing program to work up a set of ^1H - ^1H COSY data; and
2. Use the resulting spectrum to help identify an unknown.

Software

As with Lab 1, I have written these instructions to explain ACD/NMR Processor for PC. If you use a Mac, iNMR will analyze 2D NMR data, and you can feel free to use that program instead. MNova Lite isn't able to process 2D NMR data, so you won't be able to use it for this Lab.

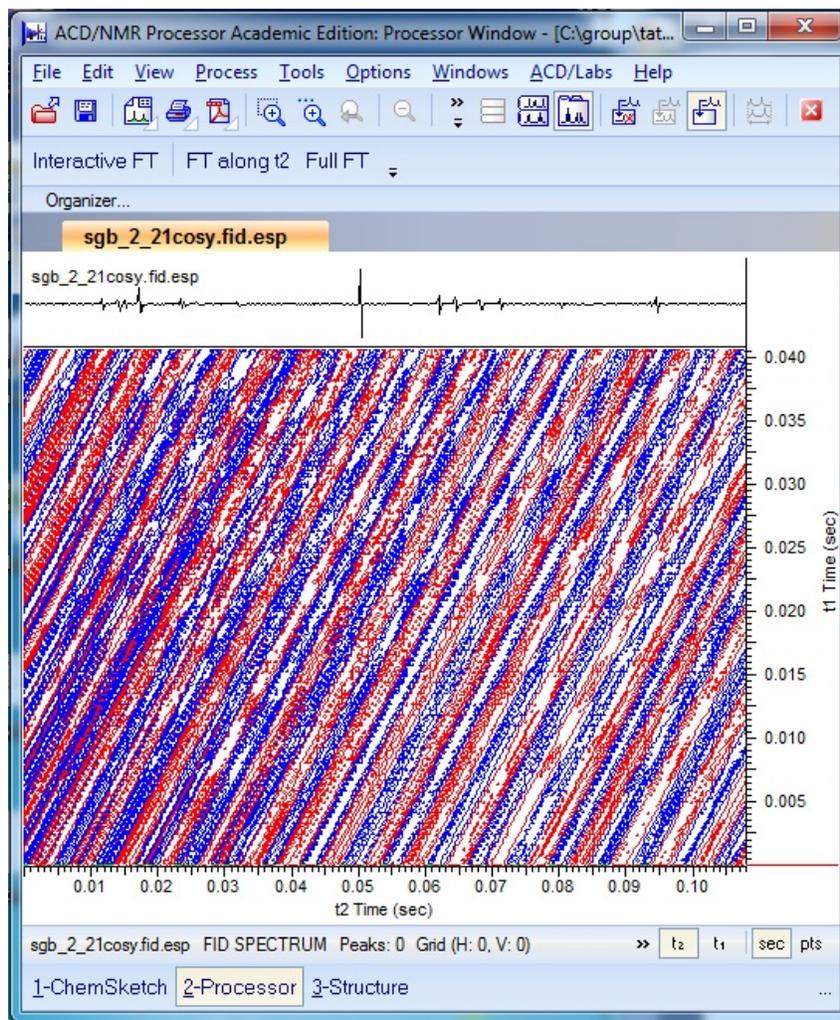
You will need to download two .fid folders to do this Lab:

- http://www.chem.umn.edu/groups/taton/chem8361/Labs/Lab3_1H.fid.zip, which contains 1D ^1H NMR data for the unknown; and
- http://www.chem.umn.edu/groups/taton/chem8361/Labs/Lab3_COSY.fid.zip, which contains 2D COSY data for the same molecule.

Processing a 2D Spectrum

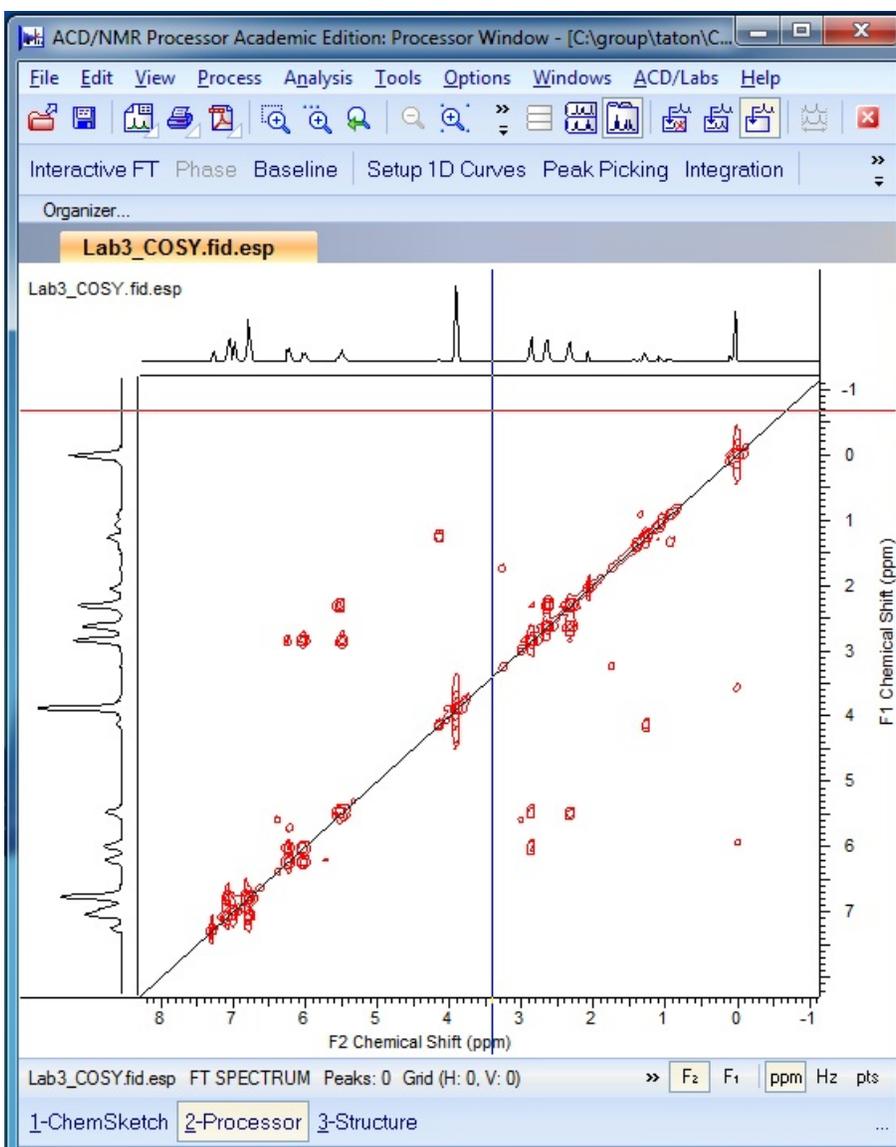
1. Before loading the 2D COSY data, you will want to load and process any 1D spectrum that relates to your 2D experiment. In this Lab, you will be analyzing a ^1H - ^1H COSY spectrum, so all you need is the 1D ^1H spectrum. If you do ^1H - ^{13}C heteronuclear correlation spectroscopy (e.g., HMQC or HMBC) in the future, you will need to process both ^1H and ^{13}C spectra. Reference, phase, and apply any FID treatment to the spectrum you need. (You don't need to do peak picking or integration; these won't show up on the 2D spectrum.) Then, in the **File** menu, **Save** the spectrum in the native ACD/NMR format (as an .esp file), and **Close** it.

- Next, **Open (From 2D File Directory...)** the *fid.dat* file from your COSY folder. Remember to set the “Files of type” dropdown box to “Any File (*.*)” to make sure you can see the *fid.dat* file in the file list.
- The program window will show a two-dimensional FID:



The easiest thing to do from here is to select **Full FT**, and to accept the default FT settings by clicking **OK** in the subsequent window. The result will be your 2D spectrum. Things to do once you get there:

- Control the vertical threshold/range either by using the mouse wheel, or by inputting threshold values into the threshold boxes. (If you can't see the boxes, expand the window horizontally until they appear in the control bar.) Your first time playing with this, you might want to set the threshold all the way down first to see the spectrum floor, and then bring the threshold up from there; you can see that some peaks disappear before others, corresponding to stronger/weaker correlations. You can also do this to get rid of crosspeaks that don't correspond to any resonance in your original 1D spectrum.



- Use the zoom buttons to focus in on a particular area and back out again.

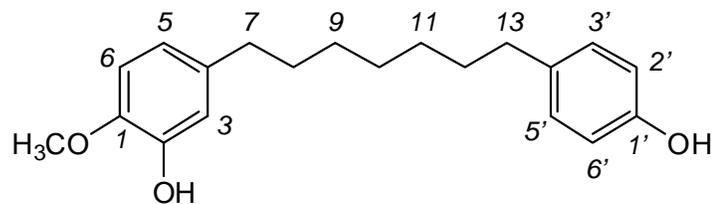
Some of the functions that you know from working up 1D data—including peak picking, integration, etc.—are all here too. But they don't work as intuitively, or mark up your spectrum as clearly, as their 1D counterparts do.

- Initially, the two axes of the 2D spectrum will be displayed either with a 1D spectrum “sum”, a sum of all intensities at each axis point through the spectrum, or a “projection”, which is just a translation of the diagonal onto to x - and/or y -axis. Because 2D NMR is typically run at higher speed, and thus lower resolution, than 1D NMR, it's likely that the 1D sums or projections look blobby. (Like in the spectrum above.) To replace these with a high-resolution 1D NMR, click **Setup 1D Curves**, then **Spectrum**. The program will ask you to find the .esp file you saved in step 1.

5. Print your spectrum, and any close-ups you like, with **File** → **Print**. Or paste the spectrum into another document with **Edit** → **Copy to Clipboard**.

Assigning Your Spectrum

Susan Brown (Hoye group) synthesized a molecule with the basic backbone shown at right; the ^1H and ^1H - ^1H COSY NMR data in this lab come from her molecule. She purified her molecule via column



chromatography using ethyl acetate/hexane as the mobile phase; her product may have been contaminated with residual amounts of either or both of these solvents. In Susan's molecule, there was unsaturation (multiple bonds) in the alkyl chain connecting the two aryl rings. In this Lab, you will determine where the multiple bonds are in the chain, and if there are alkenes, whether they are *cis* or *trans*.

Report Instructions

Each student should turn in their own report.

1. Turn in at least two printouts of the COSY spectrum: one full-range view of the entire spectrum, and at least one closeup that helped you make assignments. The diagonal does not have to appear in your closeup.
2. What was the structure of Susan's product?
3. Turn in a table assigning ^1H chemical shifts to each proton in your proposed structure.