User's Guide to ^{1}H, ^{13}C NMR

- Note: the default mouse button is the left button. Always use the left one unless told otherwise.
- For help, see Dr. Jasperse, Hagen 411-I.

1. Login
   a. double click on **jasperse** icon (or type in "jasperse")
      (Research Users: You should use your boss's login.)
   b. Password "**chem355**" (need underline in between)
   c. double click on "**xwinnmr**"

2. Sample Insertion/Lock/Tune
   a. remove cap from spectrometer if needed, and then click **LIFT ON/OFF** key on upper lefthand corner of SCM keyboard (to right of computer) to lift lock sample
   b. place your sample in sample holder, adjust position using depth gage, and place in spectrometer [DO NOT PUT SAMPLE IN WITHOUT THE SAMPLE HOLDER! YOU WILL BREAK YOUR SAMPLE AND WRECK THE INSTRUMENT!]
   c. click **LIFT ON/OFF** key on SCM keyboard to lower sample

3. Acquiring the spectrum
   a. type "**xmac**"
      - a listing of suggestions will come up
      - at present, all of these assume $CDCl_3$ as solvent
      - instructors/researchers, these can be easily customized for you needs. See Jasperse
   b. select the experiment of interest, normally **ah1-tune** or **c13-tune** or **c13-notune**
      - Note: if you are going to run both ^{1}H and ^{13}C on the same sample, you don't need to tune twice. Run "ah1-tune" first for hydrogen, then "c13-notune"
   c. when asked for file name information, type your name into the name box and
   d. type "**chem355**" into the "user" box.
   e. click **SAVE**
   f. click **COPY ALL** when the box comes up
      - The computer will now do everything for you: read in the correct parameters, lock, tune if specified, adjust the receiver gain, acquire the spectrum, phase the spectrum, and store the phasing information for automatic integration.
   g. When "ns = 8 (or 128)" box pops up, hit return to accept default, or else enter something different for the number of scans
   h. wait patiently until either an "**xmac:finished**" or "**abs finished**" message appears
      - Hopefully this whole process will take less than 5 minutes for proton or less than 8 minutes for a carbon spectrum. If 8 minutes have passed and still incomplete, see Jasperse...
   i. click on the  icon (upper right corner of the icon group) to adjust the vertical scale of the viewed spectrum. (For example, if your baseline looks flat, this will fix it!)

4. Plot
   a. click **PLOT** icon
   b. hit return in response to any boxes that appear
   c. To do **horizontal expansions**, **manual integrations** or **vertical expansions**, see instructions on page 2.

5. Exiting:
   a. Replace your sample with the default sample, as described in part 3.
   b. Type "lock cdcl3"
   c. type "exit"
   d. Say OK if it asks you anything about closing things
   e. put cursor outside of any boxes into the blue area, then press the *right* mouse button, click **Logout** and click **Yes**.

1. Plotting **Horizontal Expansions**
   a. Make sure that the cursor is somewhere on the spectrum.
   b. Click the **left mouse button**. You will now get a doubled arrow.
   c. Move the doubled arrow to the left end of the area you want to expand and click the **center mouse button** do define the **left boundary**.
   d. Move the doubled arrow to the right end of the area you want to expand and again click the **center mouse button** to define the **right boundary**.
   e. click **PLOT** icon
   f. To get back to the full expansion, click the  icon
   g. To get out of the "doubled arrow" mode, click the **left mouse** button

2. **Manual Integration**
   a. click **INTEGRATE**
   b. define the regions of interest (see horizontal expansion instructions above)
   c. click **RETURN** and save your integral regions
      • Sometimes you may wish to improve the "flatness" of the integral, or you may wish to assign calibration values of your own choosing. Do the following:
   d. put the arrow within the region of your integral, and click the **<u>left</u> mouse button**. The integral under consideration will then get a star by it.
   e. click **CALIBRATE** and respond accordingly
   f. adjust the **BIAS** in order to get the left side of the integral level
   g. then adjust the **SLOPE** to get the right side of the integral level

3. Reducing the noise in noisy, dilute ^{13}C spectra. "**Power Spectrum**" .
   [<u>Do not use for 1H spectra!</u>]
   a. After getting the normal spectrum, type "**ps**"
   b. click on the  icon (upper right corner of the icon group) to adjust the vertical scale of the viewed spectrum.
   c. click **PLOT** icon

   • Note: The "ps" command can make plots look prettier, by de-emphasizing noise. It does so by squaring all signals, however, so it will also de-emphasize small peaks that are real. In addition, by changing the relative sizes of peaks, it is incompatible with integration.

4. **Vertical Expansions**
   a. Type "**cy**" and increase or decrease the default value as you see fit. Doubling will double the printed heights, tripling will triple the printed heights, etc.
   • At default, cy=14, and is set so that the tallest peak in the spectrum will be 14cm tall. Thus, if you are wanting to expand a peak that is too tall, you need to multiply the cy as needed.

5. Manual Phasing
   a. click **PHASE**
   b. Click **BIGGEST**
   c. Click **PHO**, and keep finger held down
   d. -drag, to adjust phase of biggest, marked peak
   e. Click **PH1**, and keep finger held down
   f. -drag, to adjust phase of peaks distant from biggest
   g. click **RETURN**
   h. type "<u>abs</u>" if you want integrations to be automatically printed as a result

6. Printing Titles
   a. type "**setti**" (for "set title") b. delete existing title and type in new one
   c. click **<u>save</u>** d. click **<u>quit</u>** e. type "**title**" f. choose **<u>yes</u>**