Scheerer Lab NMR Procedure & Set Up Program

For a standard 1H NMR spectrum, there are three pieces of data we would like to see: **Chemical shift (in ppm) of each peak, integration, and coupling (**e.g. doublet, J = 2.0 Hz)

1. Log in: click on jrscheerer (old NMR/computer, log in jrsche, password: Chemi$try42)
2. **e** – eject sample
   i – insert sample. DO NOT TOUCH TURBINES WITH YOUR HANDS. Use a kimwipe.
3. Enter sample name (initials, book number, page number, crude/pure, fractions in sample ie. JRSv37_crd) under start tab, in the sample name space and comments box.
4. Make sure $CDCl_3$ is solvent selected (or proper solvent that is being used)
5. Lock: go to lock, unclick the checked box next to lock, click 'find z0'.
6. Click 'gradient shim'
7. When shimming is complete and the lock is stable, ready to start experiment!
8. Make sure correct experiment is selected – either proton NMR or carbon NMR. To load the experiment, drag the correct experiment from the left-hand options bar into the window. Now, set nt (number of transients) and bs (block size) – **nt =32** and **bs = 4**
9. **ga** – start experiment
10. After the 4 iterations, you can process the data using the following commands:

    wft fourier transform and view spectra
    aph autophase spectrum
    vsadj vertical scale adjustment
    cz clear zeros for integrals
    f full spectrum (-2 ppm to 15 ppm)

    These commands can be typed all at once into the command bar:
    wft aph vsadj cz f
11. Refrence your spectrum: In in $CDCl_3$, the standard refrence peak will be at 7.26. Click ~7.26 and type **nl** to select the nearest line, then type **rl(7.26p)** to set that as your reference line at 7.26 ppm. You can also reference the TMS peak at 0 ppm.
12. Integrate your spectrum. Turn on integral (if not already on) and use the cutting tool to chop up into appropriate pieces. Choose a peak with known integration area and set its integration value by going to the integration tab, and entering the number where it says integral area.
13. Set the size of the spectra by typing **sp=-0.5p wp=9.5p vsadj** → jrsstd
14. Print out the spectra by clicking auto plot preview (to preview what you are going to print) and then clicking print. FOR NEW NMR: print parameters in

on left tab
Click Shim
then

integration
text box

full

horizontal box – click drop down menu by display parameters and select horizontal box.

15. SAVE YOUR SPECTRA. Type **svf('samplename')** to save.
16. Eject your sample (**e**) and insert (**i**) the standard $CDCl_3$.

Carbon Spectra:
Drag and drop the ^{13}C experiment from the left hand options bar into the window. Begin experiment as described above in the proton procedure. Carbon spectra take significantly longer to aquire, especially for more dilute samples.

Carbon spectra do not need to be integrated. However, the values for number of transients and block size changes to **nt=3200** and **bs=4**. Use the same commands and procedures as outlined in the proton instructions to save and print your spectra.