^{13}C Operating Instructions for the Old Software (2006)

(Currently for INOVA 400, INOVA 600, Mercury 300)

Taking A Carbon NMR

1. Record your name, research group, spectra to be taken, and login time in the login book.

2. Load your sample, lock and shim it. (See Locking and Shimming Instructions for old software)

3. Type **C13** to load the standard parameters for a 1D ^{1}H spectrum in $CDCl_3$. If your solvent is not deuterated chloroform, type **C13('***solvent name***')** to load the parameters for that solvent.

4. Type **nt=x** where x = the number of scans you want to take. For carbon this number will need to be large, frequently greater than 100. Dilute samples may take more scans, sometimes even several thousand. To check how long the experiment will take, type **time**.

5. As carbon nuclei relax more slowly than do protons, you may need to increase the delay time by typing **d1=**X where X = 3, 4 or even 5. This will increase the time given for the nuclei to relax. For most carbon spectra **d1=2** or **3** is adequate. Increasing d1 will increase the time the experiment will take.

6. Type **ga** to begin taking scans. You may check the progress of the NMR at any time by typing **wft**. If you are satisfied with your spectrum before it is complete, you may stop it at any time by typing **aa** (abort acquisition). The scans you have taken up to this point will remain. Otherwise simply wait until the acquisition is complete.

7. Once the acquisition has finished, type **wft** to work the Fourier transform then **vsadj** to adjust the peaks to the screen. You can adjust the spectrum height at any time using **vsadj**.

8. Type **aph** to automatically phase the spectrum. Occasionally the auto phase command does not work well. In these cases you must phase manually by clicking on the **phase** button, then clicking on a peak and using the left and right mouse buttons to phase your spectrum.

9. You next need to set the reference. Press the button called **dscale** to display the scale on screen. People usually use the solvent peak as a reference, but you may use an internal standard if you wish. To see the reference, place the cursor close to the solvent peak and type **nl** to move the cursor precisely on top of the peak. Next type **rl(**XX.X**p)** in order to set the exact chemical shift of the reference peak. You can find a list of solvent peak values near every NMR.

10. You may expand on a specific region of your spectrum by clicking with the left mouse button to set one cursor, then clicking with your right mouse button to set a second cursor. This will form a box around an area of your choosing, which you can zoom in on by pressing the **expand** button. To return to the full spectrum click on the button called **full**. Alternatively, you can expand on a specific region by typing **cr=**XX.X**p** to set the left cursor value, and then **delta=**XX.X**p** to set the right cursor X ppm away upfield from the left cursor, then press **expand**.

11. You may label your spectrum by typing **text(‘***spectrum name***’)**.

12. To print, first type **vp=12** to adjust the spectrum to a vertical height the printer will recognize. There are then a variety of printing commands.

Use any commands you wish, but they should be entered in a single line, with **page** as the last command.

Example: **pl pap pir pscale page**
(this will print the spectrum, the parameters, the integrals and the scale)

a. **pl** – prints the spectrum
b. **pap** – prints acquisition parameters
c. **pir** – prints integrals
d. **ppa** – prints an abbreviated acquisition parameters list
e. **pltext** – prints text label
f. **pll** – prints peak frequencies as a list
g. **ppf** – prints peak frequencies above each peak
h. **pscale** – prints the scale
i. **page** – sends print commands to the printer

13. It is recommended that you save most spectrum. You must first create a directory, you can do this by typing **mkdir(‘***directory name***’)**. If the directory you want to save in already exists, you may access it by typing **cd(‘***directory name***’)**. To save the spectrum type **svf(‘***spectrum name***’)**.

14. In order to retrieve a saved spectrum, you must first access the directory you saved it to in the same way as above. Click the **file** button, highlight the spectrum you want to view, and click **load**. Then type **wft** and your spectrum will appear on the screen.

15. When you are finished, re-insert the standard $CDCl_3$ sample, lock it and shim it (see Locking and Shimming instructions for old software). Then type **h1**, then type **exit**. Then right click on the desktop and select **logout**. Make sure you completely logout.

16. Make sure to record your logout time in the logbook.