

The use of routine automation using organic chemistry course user id (D. E. Linn)

1. The instructor should sign the log and login to the Sun workstation using the course account and password.
2. The carousel should be removed for loading samples. Loading the tubes in place can be done but this must be done with extreme caution. Loading of samples is only to be done by the instructor and students specifically trained in doing this by Dr. Linn. Cleaning of the tubes and avoiding fingerprints on the turbines are both important issues requiring diligence. Each sample should be checked twice to ensure that each one is properly gauged to the correct depth in the spinner turbine.

An NMR is an extremely reliable but also extremely delicate and repairs and downtime can be costly to the department!!! Further keeping the NMR lab clean and free of clutter (dirty NMR tubes and solvent wastes) is expected. You must follow directions to maintain NMR privileges.

3. Make sure that the first position; typically '9' is recognized by the software and NMR hardware. If the carousel is in a different position use this number instead.

To set the hardware and software to position '9' the instructor types the command:

```
sethw('loc',9) loc=9
```

Next to ensure this has been correctly type:

```
loc?
```

The message line should respond with the software recognition of what the position is.

The herm2001 acquisition status window should also indicate whether the location is positioned at the correct location but this should also be verified. Contact the NMR personnel if this is not the case or if the software fails to respond.

4. To change the location to the desired location the instructor types the command:

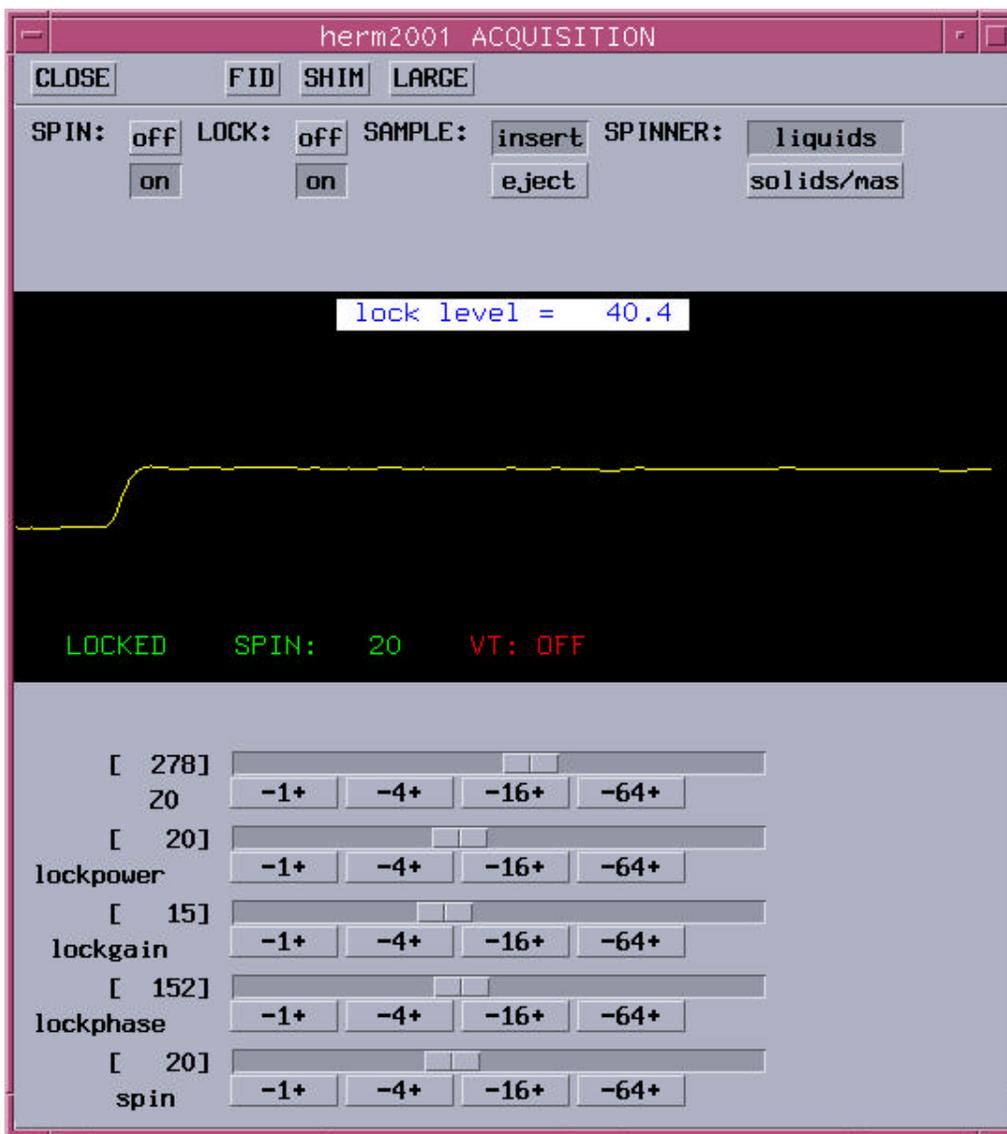
```
loc=9 change
```

If the position is already '9' the carousel goes all the way around. Don't do this. Also this step is not required if manual shimming is not necessary. Contact the NMR personnel if the hardware fails to respond correctly.

5. It is best to do manual shimming on the first sample if a set of samples with the same solvent like CDCl₃ is used. To do this select the **Acqi** button to obtain the window below. Use manual ejection only if you know what you are doing.

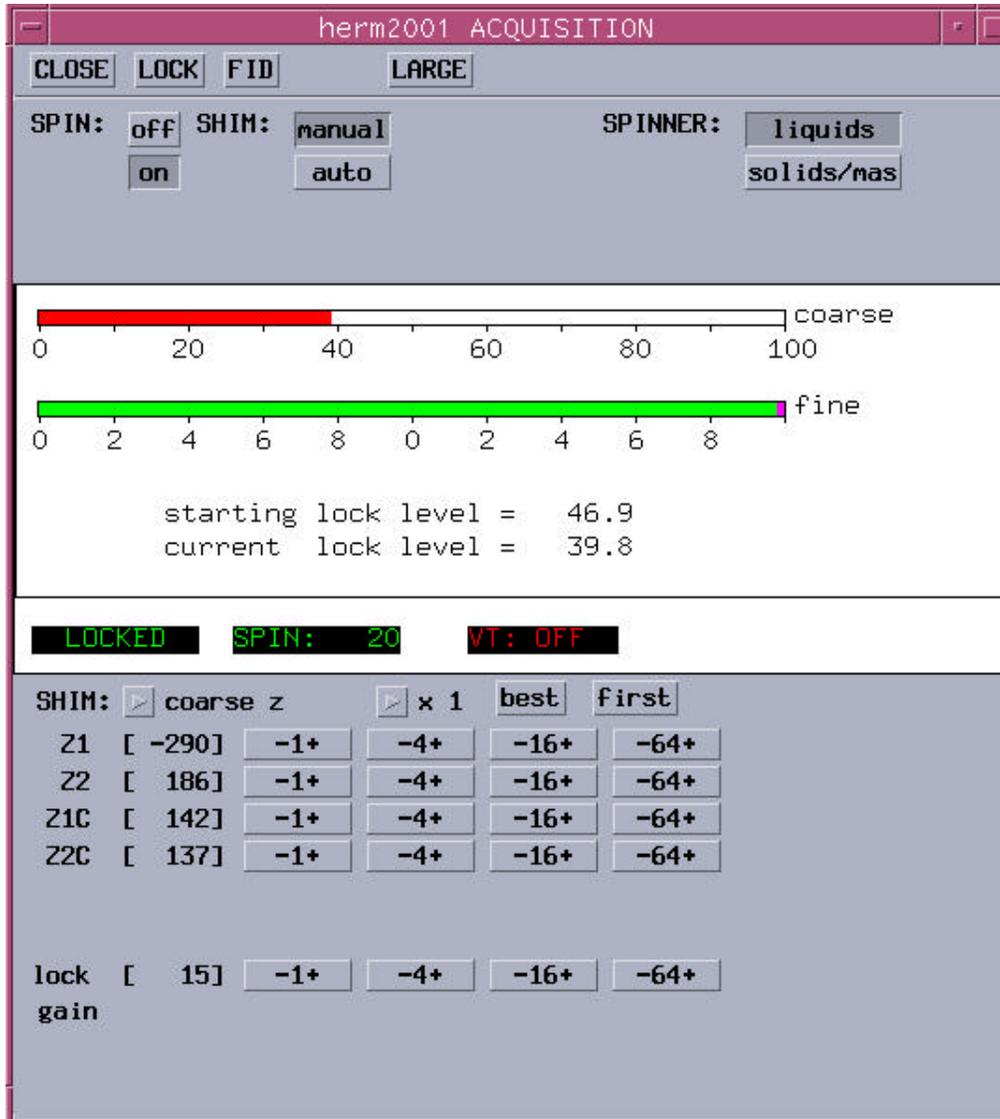


6. Next, select 'LOCK' to obtain the window that allows one to lock on the deuterium signal in the NMR solvent, typically CDCl₃.



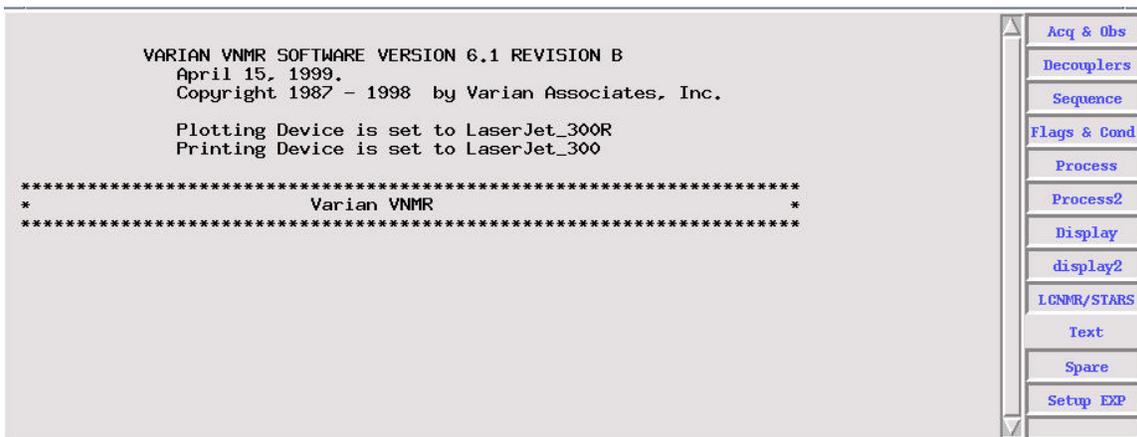
The 'LOCK' is set to 'off' initially and the main field 'Z0' is changed using the left and right mouse buttons. The condition of a match between the internal lock frequency and the deuterium frequency is desired. This condition is met when the square wave seen above is achieved. A large difference in the frequencies is indicated by a high frequency wave. The closer these frequencies are, the lower the frequency (longer wavelength) signal is observed until the above square wave is achieved. The 'LOCK' button is turned to 'on' at this point. For CDCl₃ the lockpower and lockgain should be about that shown above. See Dr. Linn if this is not the case.

7. Shim by selecting the 'SHIM' button to obtain the window below:



After obtaining lock and adjusting lock power and gain, shim on the sample using the course Z1 and Z2 (Z1C & Z2C) and fine Z1 and Z2 only. Using the right and left mouse buttons to add or subtract from the assorted shim gradients to maximize the value of the LOCK LEVEL does the shimming. DO NOT ADJUST SHIMS OTHER THAN Z1C, Z2C, Z1 and Z2 unless you know what you are doing. **If there are problems obtaining lock or shimming see Dr. Linn.**

8. To set-up an experiment use the Tck/Tk window at the bottom of the screen and select the **Setup EXP** button (see below):

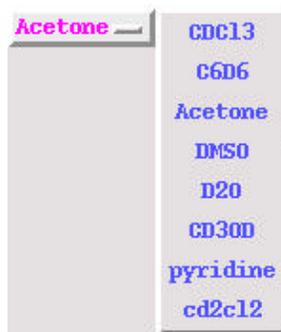


9. The automation buttons are on the left side of this new window:



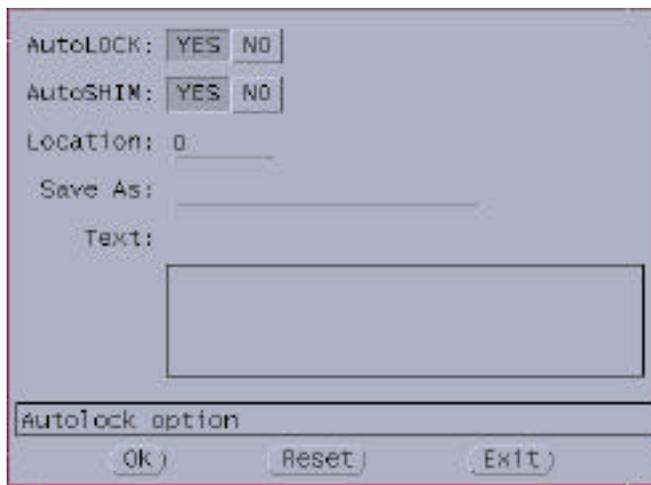
10. Do the following to run an automation sequence:

- First select the solvent from the pull-down menu by clicking on the button as shown.



- Next select the battery of experiments from the list of experiments (For example, the C13 & C13 Detected, will allow setup of the experiments CARBON plus APT/DEPT/HETCOR/PROTON/COSY/gCOSY in any combination)

11. After selecting this option the sample information window appears and must be filled out:



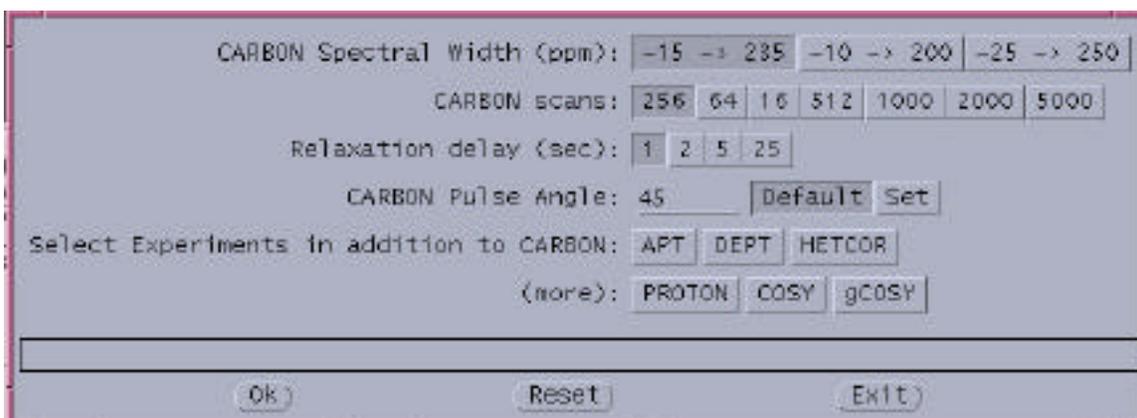
AutoLOCK: YES NO
AutoSHIM: YES NO
Location: 0
Save As: _____
Text:

Autolock option
Ok Reset Exit

Sample location, file name, and sample text information is needed here.

12. The 'OK' then 'EXIT' buttons should be selected in order when completed.

13. The window for "C13 & C13 Detected" is shown below.



CARBON Spectral Width (ppm): -15 -> 235 -10 -> 200 -25 -> 250
CARBON scans: 256 64 16 512 1000 2000 5000
Relaxation delay (sec): 1 2 5 25
CARBON Pulse Angle: 45 Default Set
Select Experiments in addition to CARBON: APT DEPT HETCOR
(none): PROTON COSY gCOSY
Ok Reset Exit

The 'OK' then 'EXIT' buttons should be selected in order when completed. Use the default (pre-selected) parameters unless directed otherwise by your instructor for each experiment. If sufficient signal-to-noise is obtained with fewer scans then only that number will be done.

14. To start automation the 'Start Acquisition' button is selected in the original Tcl/Tk window (see step 9).

15. The automation will be run on the sample in this sample location only. Printouts of the spectra will result and will have a label corresponding to the "Sample number"

16. A directory corresponding to your sample name and date will contain multiple directories for all the experiments performed and data saved.

17. Additional samples can be run when this battery of experiments is completed. A procedure for queuing samples for overnight runs is in process. Stay tuned.

18. When complete make sure that the acquisition status window indicates that the 'Decoupler' is OFF. Leaving the decoupler on will not be acceptable. It should only be on during runs and the software may not turn it off automatically so it needs to be checked.

19. Make sure when the class is finished with the instrument that it is returned to its original state. The C13 sensitivity sample should be inserted and locked (ACETONE) in position '9'.

20. Click on the gray border to bring the CDE window at the bottom of the screen to the front. Log off using the 'EXIT' button on the CDE panel at the bottom of the screen.



http://users.ipfw.edu/linn/NMR_proced_org.pdf

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