DCIF NMR Training Summary- Varian Mercury 300 MHz

The Mercury 300 is capable of observing ^{1}H, ^{19}F, ^{13}C, and ^{31}P, but is not capable of experiments such as ^{19}F decoupled ^{13}P. It is technically capable of, but not well-suited for, 2D NMR. The instrument has an 'AutoSwitchable' probe run in 4 NUC Mode. Unlike most of the other DCIF instruments, the Mercury 300 does not have variable temperature capability and is not tuneable. The Mercury is ideally suited for quick 1D spectra and is on 'walkon' status for most of the day. During walkon hours, researchers can use the instrument for limited amounts of time (see posted time limits) without scheduling in advance. The Mercury uses the same version of VNMR (currently 6.1c) as the INOVA 500 and INOVA 501. The complete Mercury 300 Training Guide can be found on the DCIF website.

Conventions used in this document:

Bold characters without brackets indicate a command to be typed into the command line.
[Bracketed bold characters] indicate buttons to be clicked on.
Italics indicate basic troubleshooting solutions for common problems.

Login, Setup, and Acquire

1. Log in to the host computer (MrHat) using your assigned user identification (UID). Click on the Spectrum icon on the bottom toolbar. Sign into the paper logbook on the desk (legibly!).

2. Load the bestshim file by typing **bestshim**. Alternatively, you may create and use your own shim files using the **svs** (save shim file) and **rts** (retrieve shim file) commands. Example: **rts('my_own_shim_file')** will load a shim file previously saved as 'my_own_shim_file'.

   *If 'setup complete' doesn't appear in the status window, or if you do not hear the computer beep, it might mean there is a connection problem between the computer and the console. You can test this by typing **su** and listening for a beep. If there is no beep, then the computer is unable to reach the console- you should type the command **reset**, wait until it finishes (~20 seconds), then try **su** again. If there is still no beep, alert a DCIF staff member or email dcifhelp(at)mit.edu so someone can come reboot the console.*

3. Properly position the sample (i.e. center the solvent volume) using the depth gauge.

4. Type **e**, listen to hear the air come on, then place the sample in the opening at the top of the magnet. Type **i** to lower the sample. You should hear one or two clicks as it is lowered into the magnet.

5. Click the **[Acqi]** button. The Acquisition Window will open. *If the **[Acqi]** button is not visible type **acqi** in the input window.* In the acquisition window, click **[LOCK]**.

6. Set the spin rate to 20 and watch in the Acquisition Status Window until you see the spinner is regulated.

7. Consult the handout by the spectrometer computer to determine the appropriate lock power for your solvent. You may wish to maximize the lock gain (amplification on the signal leaving the instrument), as this can help with the next step.

8. Adjust Z0 until the sample is on resonance (plateau shape) then click LOCK: **[ON]**. Check the Acquisition Status Window- the lock should be regulated. Lower the lock gain until the lock level is between 50 and 70. Adjust the lock phase with the **±4** button to maximize the lock level.

9. Click **[SHIM]**. Maximize the lock level by adjusting Z1C and Z2C- use the **±1** button. If your lock level nears 100, lower it by decreasing the lockgain. Z1C and Z2C are often sufficient, but you may also adjust the fine shims Z1-Z5 to get better peak shape.

10. Click **[CLOSE]**. Save your shims if you want using the **svs** command.

11. Click **[main menu] [setup]** and choose the desired nucleus and solvent.

12. Adjust the default parameters, if desired, using the formula "name of parameter =value" (e.g. **nt=8** will change the number of scans to 8)
    ~nt is the number of transients (scans) collected- it must be some multiple of 8! The default is 16 for ^{1}H and 256 for ^{13}C.
    ~bs is the block size. This parameter determines how often the data is written to disk. During long runs, this parameter allows you to monitor the progress of your run by typing **wft** (weighted Fourier transform) after each block has completed. It must be higher than 4, and the default is 16

    Other Acquisition Commands:
    dg (display group) will display the parameters you are using in the 'text window'.
    dps displays the current pulse sequence.
    sa will stop the acquisition after it completes the current scan and will allow you to view your results.
    aa will abort the acquisition immediately- you will not able to view the data.

13. Type **su**. Wait for the beep. *If there is no beep, see step 3 for reset/reboot instructions.* Type **ga** to start the acquisition. *If you get a cryptic message about an autogain failure, reduce your pulse width by typing **pw=pw/2** then doing **ga**. The most likely cause for this is an over-concentrated sample. You may have to reduce the pulse width more than once.*

Process

You may wish to save your data with **svfz** before processing. *If **svfz** doesn't work, use **svf** instead.*

Overview of Processing Commands:

aph will automatically phase the spectrum.
vsadj will adjust the vertical scale.
ds will display the spectrum again and get you out of phasing, integration or display mode. It will also fix occasional display or cursor glitches.
dscale will display a scale in ppm at the bottom of the spectrum.
ffav will automatically display the full spectrum, phase, adjust the vertical scale and display the horizontal scale for you. **ffav** is an 'in house' macro that consists of **f full dc aph vsadj dscale**.

Zooming in and out

VNMR switches between single cursor and double cursor (box) mode. You place and move the first cursor by left-clicking on the spectrum. Right-clicking will place a second cursor. If you have both cursors on the screen, a left-click will move both. The **[expand]** button will zoom in on the region between the two cursors. So to zoom in on a specific peak or region, you must first left-click on the left side of the desired area, then right-click on the right side, then click **[expand]**. To return to the full view, click **[full]** or type **f**. Hold down and drag the middle mouse button to stretch or shorten the peaks.

14. If phasing from autophase (**aph**) was not satisfactory, you can click **[phase]** to manually adjust the phasing. The left mouse button adjusts the 0_{th} order phasing, and the right mouse button adjusts the 1_{st} order phasing. When you're satisfied, use **ds** to leave phasing mode.

15. To reference your spectrum: Locate the solvent or TMS resonance. Zoom in on it, then place the cursor on top of this peak and type **nl** (nearest line). Then click **[REF]** and enter the literature value for the chemical shift.

16. To integrate your spectrum: Click **[part integral]** to display the integral line. *If incorrect integral regions appear, type **cz** to clear them.* Click **[resets]** and use the left mouse button to define the regions you wish to integrate. The right mouse button can be used to undo mistakes. When you are done, type **ds** to leave resets mode. Choose one integral area to be your reference, click on it, then click the **[Set int]** button and enter the integral value. To view the peaks as they will appear on the printout, type **dpir**, then **ds** to leave display mode.

17. To pick peaks: Click the **[Th]** button to set a threshold with the mouse. To view the peaks as they will appear on the printout, type **dpf,** then type **ds** to leave display mode.

18. To add text to your spectrum: Type **text('blah blah blah')** (single quotes required) to put in whatever text, in this case blah blah blah, you want to have displayed in the upper left corner of your spectrum.

Save

19. Type **svfz** save your data. The **svfz** command will save one copy of your data on the NMR computer, MrHat, and one copy on the DCIF's Varian data server, Zippy. *If the computer slows down after the svfz command or you do not see a message stating your data was also saved to Zippy, then it may not have saved properly. Save using **svf** instead.*

Print

20. To plot the spectrum, string together any of the following plotting commands followed by **page**:
    - **pl** will plot the actual spectra.
    - **pscale** will plot the scale below the spectrum.
    - **pir** will plot the integral amplitudes below the spectrum (defined earlier in step #16).
    - **pirn** will plot normalized integral amplitudes below the spectrum.
    - **pap** will plot out 'all' acquisition parameters.
    - **ppa** plots a more easily understood version of **pap**.
    - **ppf** plots the peak frequencies in ppm (above the spectrum).
    - **ppfhz** plots the peak frequencies in hertz.
    - **pldate** plot the current date and time in the upper right-hand corner.
    - **pltext** will plot the text you defined above (in step #18).
    - **page** will dump the plot buffer to the plotter.

Logout

21. Click **[acqi]** *Type **acqi** if the button is not present.* Click **[lock]**, then spin **[off]**, lock **[off]**, and **[eject]**. Get your sample. Click **[insert]**, **[close]**. Type **exit** at the VNMR command line. Right-click on the desktop background and logout. Please sign the logbook and note any problems you may have had with the instrument