

DCIF NMR TRAINING SUMMARY

VARIAN MERCURY 300 MHZ

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The Mercury 300 is only capable of observing ^1H , ^{19}F , ^{13}C , and ^{31}P , but due to hardware constraints is not capable of experiments such as ^{19}F decoupled ^{13}P . The instrument has an 'AutoSwitchable' probe run in 4 NUC Mode. In this configuration, it does not have the broadband capabilities of the Avance 400, or Inova 501, however it does not require the user to tune the probe. Unlike all of the other Facility instruments, the Mercury 300 also does not have variable temperature capabilities. The Mercury is ideally suited for quick 1D spectra but is fully capable of many of the more sophisticated multiple dimension experiments. The Mercury uses the same version of VNMR (currently 6.1c) as the INOVA 500 and INOVA 501. The Mercury 300 Training Guide can be found on the DCIF website.

1. Login to the host computer (MrHat) using your assigned user identification (uid). If you don't have an account (as is with all the DCIF instrumentation) access is forbidden. No exceptions. Please see DCIF staff if you would like to be trained.

restart: If you login and find the VNMR program running, a message may indicate vp doesn't exist. The previous user from your group forgot to type **exit** before logging off. Type **restart** in VNMR to close the current program, and then reopen VNMR.

2. Click on the Spectrum icon at the bottom of the screen on the CDE (Common Desktop Environment) toolbar, or right mouse-click the background and select VNMR.
3. The program will automatically change to your personal directory. You will also be prompted if you would like to load **bestshim** (answer yes or no... the choice is yours). If you select 'no', you may also load any other shim set using the **rts** command (if you have previously saved a shim file with the **svs** command).

Example: **rts('my_own_shime_file')** will load a shim file previously saved as 'my_own_shime_file'.

If 'setup complete' doesn't appear in the status window in the bestshim macro doesn't run or you do not hear the computer beep, type **reset** in VNMR. The macro **reset** will automatically stop and restart the acquisition daemon. If reset does not work, open a terminal window and type **su acqproc** once or twice (once to go from Idle to Inactive in the acquisition status window, and once more to restart the acquisition daemon).

4. Click **[main menu] [setup]** and choose the desired nucleus and solvent then type **su**. Wait for the beep.
5. Click the **[Acqi]** button. The MrHat Acquisition window will open. If the **[Acqi]** button is not visible type **acqi** in the input window. If the Acquisition window still doesn't open, perform an **su acqproc** or **reset** as described above.

In the MrHat acquisition window click **[LOCK]**.

6. Properly position the sample (i.e. center the solvent volume) using the depth gauge located near the magnet leg. Note that shimming the sample is easiest when the proper volume (0.7ml) is used.
7. Click **[eject]**, then place the sample on top of the magnet bore tube. Click **[insert]**, wait until you hear a click, then click SPIN: **[on]**. Remember that the default spin rate is 20.
8. Adjust Z0 until the sample is on resonance then click LOCK: **[on]** to lock the sample. Use the highest power you can without excessive saturation. A lock power of 26-27 is about right for CDCl_3 . Adjust the lock phase to maximize the lock signal.
9. Click **[SHIM]** to open the shimming window. Adjust the Z1C and Z2C, with the ± 1 button. If your spectrum is going to be crowded (lot of resonances) or if you want a publication-quality spectrum, adjust Z1-Z5 as well.
10. Click **[CLOSE]**. Save your shims if you want using the **svs** command.

11. The standard parameters will load the default sweep width (sw), pulse width (pw), and several other 'standard' values. Values you may wish to change:

nt is the number of transients (default to 16 and 256 for proton and carbon respectively) to collect. If you want to change nt, type **nt=64** (or any other number you may want).

bs is the block size (default is 16 for proton). This parameter determines how often the data is written to disk. To change this value type **bs=8**. During long runs, this parameter allows you to monitor the progress of your run by typing **wft** (weight, Fourier transform) after you see the message "exp1: BS 1 completed" and so on.

dg (display group) will display the parameters you are using in the 'text window'.

dps displays the current pulse sequence.

12. Type **ga** (submit the experiment to acquisition and Fourier transform the result) to start collecting the spectrum. If you get a cryptic message about an autogain failure, reduce your pulse width by typing **pw=pw/2 ga**. The most likely cause for this is your sample is too concentrated. Repeat as necessary.
13. Simple processing commands:

aph will automatically phase the spectrum. You can also click [**phase**] to manually adjust the phasing with the mouse. Left mouse button adjusts the 0 order phasing, and the right mouse button adjusts the 1st order phasing.

vsadj will adjust the vertical scale **vs**.

vs can be automatically adjusted using the middle mouse button.

ds will display the spectrum again and get you out of phasing mode.

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**.

14. To reference your spectrum:

Locate the solvent resonance or some other reference line (such as TMS). By clicking with the left mouse button on one side of the peak and then clicking with the right mouse button on the other side of the peak you can then click [**expand**] to expand this region of interest. Place the cursor on top of this resonance and type **nl** (nearest line). Now type **rl(7.27p)** (for chloroform) or whatever ppm shift is appropriate for that resonance. You can also click [**REF**] and type the shift value in ppm. The command **dscale** will display the entire scale.

15. To integrate your spectrum:

Click [**partial integral**] to display the integral line. Type **cz** to clear all resets (zeroes) already in memory. Click [**resets**] and use the left mouse button to define those regions you wish to integrate. The right mouse button can be used to undo mistakes. The parameter **is** is the integrals scale and can be adjusted using the middle mouse button while in integral mode.

Type **ds** to regain cursor control. Place the cursor over the desired region and click the [**Set int**] button. You will now be queried for an integral value.

Type **ds dpir** (display peak integral regions) to get the integral values displayed underneath the spectrum.

16. To perform peak picking:

Click the [**Th**] (threshold) button to interactively define a threshold with the mouse.

dppf will display the peaks in ppm.

dpphz will display the peaks in hertz.

Type **ds** to erase the displayed 'peak picking'.

17. 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 hand corner of your spectrum. To plot just the text, use the macro **plttext**. Other positioning options are available. Type **man('plttext')** to learn more. Another option is the macro **getttext**. This will open a small easy-to-use text editor, which will easily allow you to place text on multiple lines.

18. 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 #15).

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.

plttext will plot the text you defined above (in step #17).

page will dump the plot buffer to the plotter.

Example: **pl pppfhz pscale plttext page** will plot the spectrum, peak positions (in Hz), the reference scale, and text.

19. Type **svfz** save your data to a file. The **svfz** command will also save a copy of your data set to Zippy in the sub-directory 'mrhat' of your home directory. If the computer slows down after the svfz command, Zippy may have crashed or the network may be down; use svf to save your data on MrHat only. The command **rt** will allow you to retrieve a file. You should already be in your home directory, but if not, type **gohome**.
20. Click **[acqi]** (type acqi in the Input Window if the button is not present). Click **[lock]** spin **[off]** lock **[off]** **[eject]**. Get your sample. Click **[insert]**, **[close]**. Type **exit** at the VNMR command line. Click on EXIT on the CDE toolbar and confirm the logout. Please sign the logbook and note any problems you may have had with the instrument.