SHORT OPERATIONS MANUAL
FOR THE MERCURY Vx NMR with VnmrJ 2.2D

Log into the system by typing your name at the login prompt. Enter your password when it is requested. After you log in, you can start the NMR software by clicking the VnmrJ icon on the screen.

The computer operating system for this software is Linux. This means that all commands are case sensitive and should be entered in lower case.

The screen is divided into a number of panels. These panels, except for the spectral window, can be open or closed using the push pin on the side of the panel. This will hide the panel and place a tab at the edge. If you move the mouse over the tab the panel will open again. The panels can be totally closed by clicking the X. However, the only way to re-create them again is to click ***View*** at the top and choose the panel you wish to open

Entering Commands and Parameters

Although this software is almost totally run using icons if you are familiar with the older version of Vnmr you can still use the command line. If this line is not open at the top of the spectral window it can be found by dragging the top line of the spectral window down to expose it. If you continue dragging that line down you will see a list of the previous commands that you type during your present session.

Entering a command can be carried out by clicking the appropriate icon or typing the command followed by a return. A number of commands can be entered in the command line at the same time as long as they are separated by a space.

Changing the value of a parameter can be done by finding the appropriate menu in which to enter it or by typing, in the command line, the parameter followed by an equal sign and then the new number, ie ***nt=64.*** If the parameter is a text value ie. ***y*** or ***ynn*** and you wish to enter it using the command line, it must be surrounded by single quotation marks, ***dm='ynn'***.The value of any parameter can be checked at any time by typing the parameter followed by a question mark, ie ***nt?***.

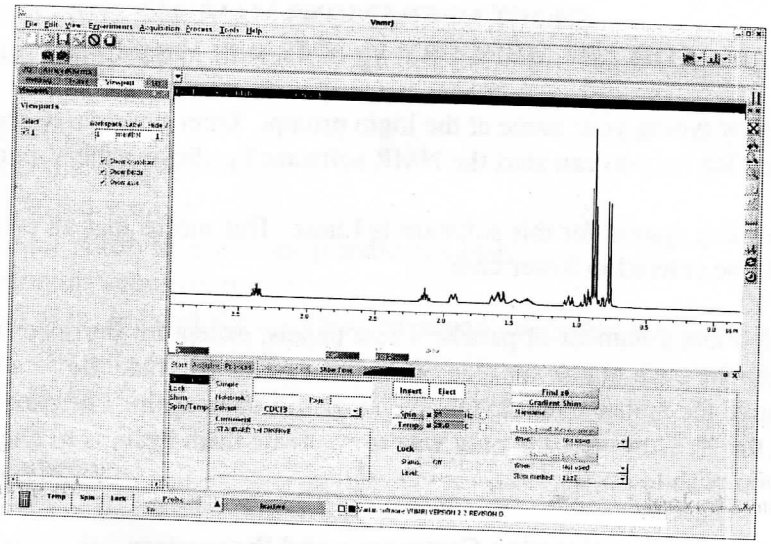
Choosing the experiment

Click ***experiment*** on the top line and choose the experiment that you wish to run from the drop down menu.

The parameter panel below the spectrum will now have a number of buttons that will help you setup your experiment, collect your data and process it.

Click on ***start***

Down the left side of the parameter window, below the spectrum, will be a few new buttons. Click on ***standard***. In this window you can enter a filename (Sample), notebook information, comments and the solvent. You choose the solvent by clicking the arrow next to the solvent and selecting the correct solvent. If you do not enter the correct solvent your spectrum will not be correctly centered.

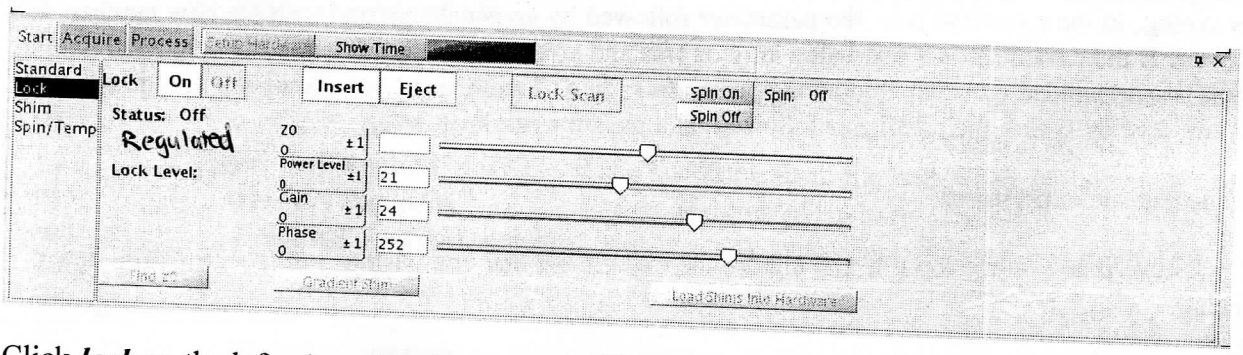


Click ***eject*** to remove the previous sample and ***insert*** to insert your new sample. You can check if the sample is spinning by looking at the spin parameter at the bottom left of the window and making sure it is not 0.

Locking

You next need to lock the sample to prevent drift during the data acquisition. This is done by clicking the button that says ***find z0.*** If you are having trouble with this use the manual locking procedure described below. Please follow the directions carefully.

Manual Locking on Mercury Vx



Click ***lock*** on the left edge of the panel.

turn lock ***off***.

Set lock phase to approximately

set lock gain and lock power to maximum. This can be done using the sliders

Click ***Lock Scan*** to see the lock window.
You should see some wiggles in the lock screen.

Use the Z0 control to reduce the frequency of the wiggles as much as possible. You may have to increase or decrease Z0. If you often use the same solvents make a note of the Z0 positions so that you can set it directly.

Turn lock ***on***.

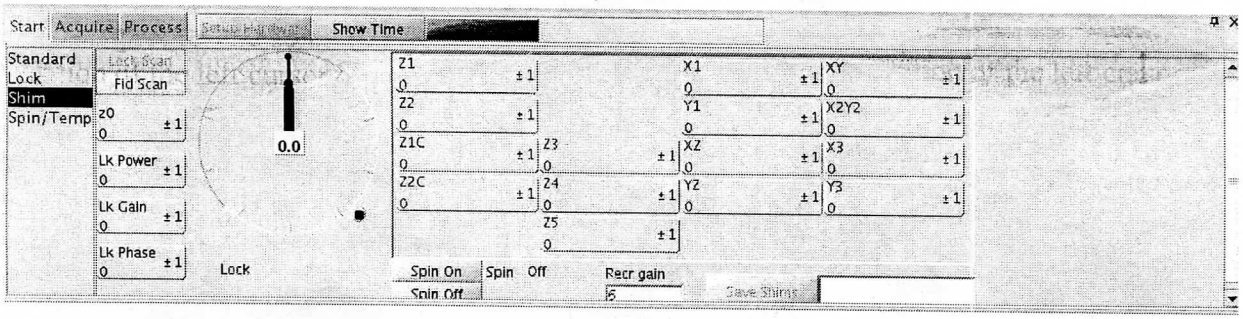
Reduce the lock power and lock gain level to obtain a lock level of approximately 50. Try to keep lock power lower than lock gain.

Then shim as usual.

Shimming

Once the sample is locked you now have to shim the sample to get the highest possible resolution. Make sure that the sample is spinning. If it is not spinning click ***spin on.***

Click on ***shim*** along the left side of the window. The shimming screen contains the lock level indicator which shows the lock level as a clock where the hands indicate the intensity of the lock signal, the lock power, lock gain buttons and the shim buttons.



Although all of the shim controls are shown, only adjust the Z1C, Z2C. If you think you need some better shim values then also adjust Z1 and Z2 . The shims are adjusted by clicking on the appropriate shim button with the left mouse to make the shim value decrease or the right mouse button to make it increase. If you should happen to click the middle mouse button you will notice that the multiplier in the lower right corner of the button will change. Please keep this value to 1. If it is not 1 you can cycle through the values using the middle mouse button.

The goal of shimming is the make the lock level as high as possible! There is no specific answer!

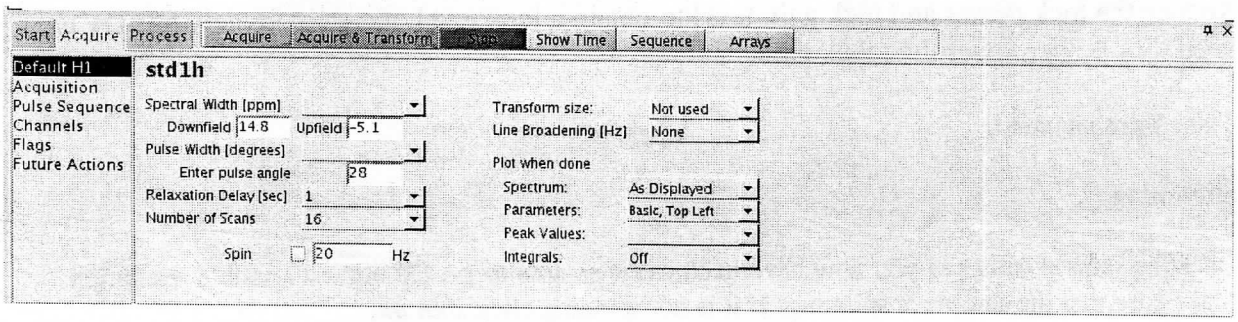
Start with the Z1C shim. Change the value by +1 unit to see the effect. If the level increases continue changing ZIC by +1 unit until the lock level in maximized. If the level should decrease change the shim value in the other direction. Continue clicking the left or right mouse buttons to maximize the signal. Do the same thing with the Z2C shim. Go back and forth between the Z1C and Z2C shims until the signal is maximized. If the lock level should go off the scale (>=100) you reduce the lock gain so that the lock level is on the screen. Alternatively you can change the lock power in a similar fashion.

Once the sample is properly locked and shimmed you can now collect your data.

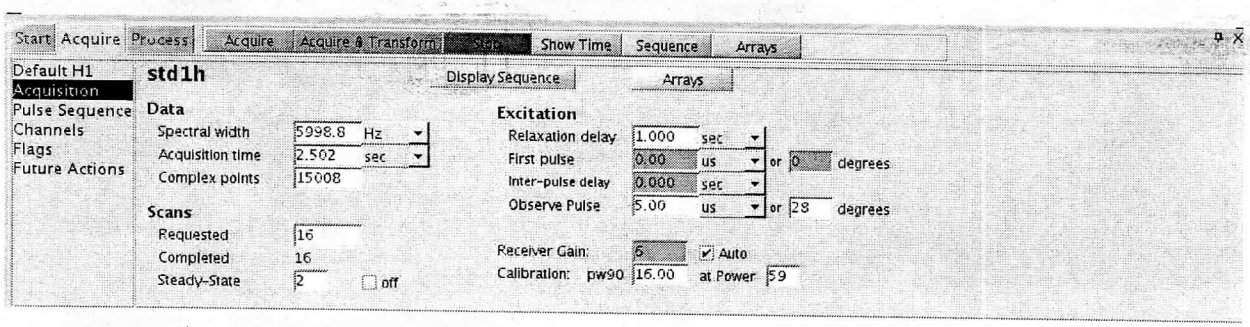
Click the gray ***acquire*** button along the top of the parameter window.

Collecting data

Under ***Default*** you will see a list of standard parameters. You can change these by clicking on the arrows and selecting default values. Unfortunately, in this window you are restricted to only the default values.



If you wish to use values other than the defaults you must go to the ***Acquisition*** window. In this window you can change the values to anything that you want. Only change the values that you understand. These include the spectral width, the number of scans and the relaxation delay. Do NOT change any other parameters unless you know what you are doing.

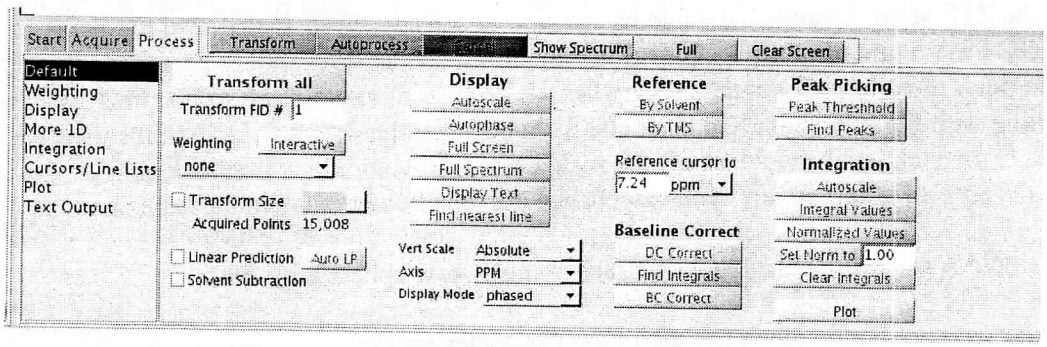


Once these values are set you can start your data collection by clicking either ***Acquire*** or ***Acquire & Transform***

Manipulating the data

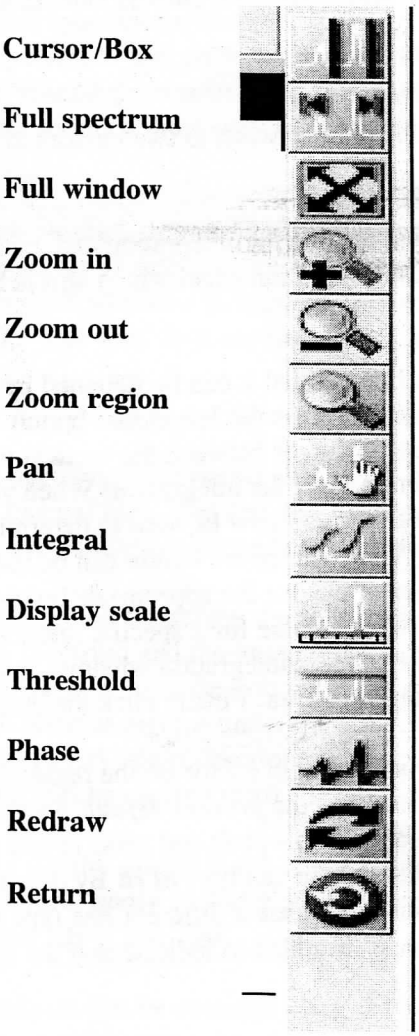
After the data is collected the spectrum will appear on the screen. Along the right edge of the spectrum will be a tool bar that has similar functions to the button bar in Vnmr. These buttons are shown below.

Click ***Process*** and ***Default***.



Phasing the Spectrum

In most cases the spectrum will need to be phased. The simplest way to do this is click ***Autophase*** in the Display menu. If this does not do a satisfactory job click ***phase*** in the tool bar. Put the mouse over some peaks on the right side of the spectrum about half way up the screen and click the left mouse button. There should now be two vertical red lines and a horizontal red line. The horizontal red line is the zero position. Put the mouse so that it is between the two vertical red lines and on the horizontal red line. Hold down the left mouse button and move the mouse up or down to correctly phase that portion of the spectrum. Now click the mouse over some peaks on the left side of the spectrum half way up the screen. You should again see two vertical lines and a horizontal line. Phase this portion of the spectrum. When this is completed, repeat the procedure on the right hand part of the spectrum. Go back and forth between the left and right hand sides of the spectrum until you are satisfied with the spectrum phase then click the ***cursor/box*** menu button.



Displaying and Plotting the Spectrum

The vertical size of the spectrum can be changed using the middle mouse button. Placing the arrow above the baseline and clicking the middle mouse button increases the height of the spectrum. Doing the same with the arrow below the baseline decrease the spectral height. If you put the arrow on or above a peak and click the middle mouse button the peak will go to that height.

Displaying and manipulating the spectrum is carried out using the mouse and the process tool bar.

box/cursor*:** This allows you to manipulate the cursors. It will toggle between ***box and ***cursor*** when clicked. When there is one cursor on the screen this can be moved by holding down the left mouse button and moving the mouse. Clicking the right mouse button will put up the second cursor. This can be moved by holding down the right mouse button and moving the mouse. If there are two cursors on the screen holding down the left mouse button will move both cursors simultaneously.

full: Returns the spectrum to the full width.

zoom in/zoom out These are used to expand the area between to two cursors to the full window or return to the full spectrum.

part integral/full integral/no integral: This will turn on the integration and expand the tool bar to include ***lvl/tlt*** and ***resets***. Also go to the process integration panel which will help you carry out your integration.

You will now see a green integral line. If this line is tilted it can be flattened by clicking on ***lvl/tlt*** icon. Put the cursor near the left end of the spectrum and press the left mouse button. There will now be two vertical red lines and a horizontal red line. Put the cursor between the vertical lines and hold down the left mouse button. Move the cursor up or down to level the integration. When you are finished you can set reset points by clicking ***resets***. The mouse arrow can now be used to determine the position of the reset points by clicking the left mouse button. The right mouse button can be used to remove reset points. To remove all of the reset points type ***cz*** or choose the appropriate button in the panel. When you are finished click on the ***cursor/box*** button. To set a value for a specific integral put a cursor through the integral that you wish to set. Then go to the process/integration window. In this window there is a place to enter the number of protons in the integrated area. Finally click the button that says set integral.

Setting the Reference: To set the reference place a single cursor on the residual solvent peak or any other peak that you want to define as the reference. In the ***process/default*** window click ***find nearest line***. Enter the chemical shift that you want to use for this peak and press return and the peak will now be calibrated to the chosen frequency. Alternatively you can type ***nl rl.*** By default the instrument assumes that the reference line is 0 ppm. If you want to use a different line type ***nl rl(x.xxp)*** where x.xx is the chemical shift of the chosen peak and the 'p' is added to indicate that the number is in ppms.

Expansion

Expansions of specific areas of the spectrum can be carried out so that they can appear on the same sheet as the spectrum or be plotted on a different page. Before beginning your expansions it is recommended that you save your initial plotting parameters so that they can be

recalled later. This can be carried out by typing ***s1*** which saves the plotting and screen parameters to the first save location. There are nine such location that can be accessed by typing ***s1, s2, s3....S9.*** Type ***r1 r2,r3....r9,*** to retrieve the corresponding set of parameters. To expand a section of the spectrum to another page, place the cursors around the area you wish to expand. Click ***expand*** and that section will be expanded on the screen. If you wish to have a specific spectral window the start of plot sp (***sp=xx.xp***) and the width of plot wp (***wp=x.xxp***) can be set manually (note the width of plot is the total width between where you want to start and where you want to end and may not be the position of the end of the screen). If you wish to locate the peak positions on the spectrum click the ***threshold*** button on the tool bar to put up a threshold and adjust it to the height you wish using the mouse and the left mouse button. All peaks above the threshold line will be plotted on the spectrum. To see which peaks have been chosen type ***dpf*** (display peak frequencies). If you are satisfied then type ***pl ppf*** to plot the spectrum with the peak positions listed on the chart in ppm. If you wish the peak positions to be in hertz type ***axis='h'***or find the appropriate button in the menu that does this. This will also convert the scale to hertz. If you want the scale back in ppm you must type ***axis='p'***.

To expand a section of the spectrum on the same page, place the cursor close to the area you wish to expand. Type ***inset.*** The enclosed region will appear above the area designated. This area can be moved around the screen using the ***sc wc*** button where the left and right mouse buttons are start of chart (sc) and width of chart (wc) respectively.

Place the spectrum where you would like it. In order to change the vertical position you must type *vp=* and the position in mm from the baseline. If you wish to plot the spectrum with the peak positions indicated follow the procedure described above. The computer now has the inset display parameters in its memory. If you wish to expand other regions you must first return your initial display parameters by typing ***r1***. You may then expand another region in the same fashion as described above.

Plotting

Plotting can be carried out using the ***Process plot*** panel. Here you can choose what you want on the printout and then click manual plot. You can also click autoplot which will give you the standard plot. You could also type in the standard commands listed below.

To plot the spectrum you see on the screen the following list of commands will be used:

pl	plot the spectrum and the integral if it is on the screen.
pscale	plot a scale in ppm at the bottom of the page.
pir	plot the integral regions under the scale.
ppa	plot parameters (short description) or ***pap*** plot paramaters (long description)
page	**Nothing will be plotted until the page command is given.**

The plot parameters are usually placed in the upper left hand corner of the paper. To move them to another location use the form ***ppa(x,y)*** where x and y are the locations on the paper in mm. The lower right is 0,0 and the upper left is 280,200.

A listing of the line positions can be obtained using the ***pll*** command after a threshold has been set. It is suggested that it be put on a different page so that it will not write over your spectrum. All of the above commands may be strung together separated by spaces to save typing and time.

Saving your Data

Data may be temporarily saved to the hard drive by typing ***svf***. The system will then ask for a file name. Type in a file name. Do not use any spaces or special characters in your file names. Also do not use any dot three extensions because the computer will add .fid to the end of the file name.

Exiting the computer

To exit the nmr software click file in the top menu and then exit from the drop down menu. You must also remember to exit from the computer. In order to do this click on the system button at the top of the window then click logout in the drop down menu. Be sure that you see the login screen before you leave the room.

Saving Data to Your Prism Account from the Varian NMR's

Anytime that you wish to transfer your data you must be in a terminal window. This can be opened on the Varian instruments by clicking the right mouse button in the window. A menu will appear. Click **open terminal**. A terminal window will open. You now need to be in the directory that contains your data. In most cases this will be in your current directory. You can check this by typing ***ls*** which will list the files in the current directory. If you see your data files you can continue.[1] To transfer the data type the command below:

scp -r <filename> <email address>@scp.prism.gatech.edu:

The ***filename*** is the name of the fid file that is in your directory. Type in the name exactly as it appears in your directory and include the ***.fid*** at the end. The ***email address*** is your personal Georgia Tech email address on the Georgia Tech prism computer. The ***: (colon)*** at the end of the line is very important so do not forget to put it in.

The first time that you do this you will be asked a yes or no question, answer yes.

You will then be asked to enter your password. This is your **email password** and not the NMR password.

While that data is being transferred you will see some information going across the screen indicating the transfer. If you do not see these lines it might mean that you forgot to put the **:** (colon) after the line. After the data is transferred you can remove the data from the hard disk or you can leave it until you are sure you have properly transferred your data. If you wish to remove your data you can type the following:

rm -r filename

If you want to remove all of your NMR files then use the following command which contains the wildcard character * which means any characters of any length.

rm -r *.fid

This will remove all files that end in ***.fid***. The * can be used to remove any specific group of files. If you wish remove all NMR files that begin with the letter g you can type

rm -r g*.fid.

Wildcards can also be used to transferring your data. To transfer all files that begin with g type:

scp -r g*.fid <email address>@scp.prism.gatech.edu:

After you are finish you may want to keep the terminal on your desktop for future reference. This works the same way as on a pc. If you click the small square in the upper right hand corner the terminal window will collapse to an icon. Double click on the icon to reopen the terminal window.

1. If you do not see your data files when you type ***ls*** it means that they are in a different directory. The most probable place is in the data directory under vnmrsys. It order to get to this directory check the current directory list for ***vnmrsys***. If it is there type

cd vnmrsys/data

Then type ***ls*** to see if your data is here. If it is not contact me I will help find your files.

Questions About Advanced Operations.

If you have any question about the system or need to carry out any advanced experiments, ie. variable temperature, T1, T2, COSY, NOESY or anything else please contact Leslie Gelbaum for assistance at 404-894-4079 (office), 404-894-1827 (lab).