

DCIF NMR TRAINING GUIDE

VARIAN MERCURY 300 MHZ

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This document is intended as an introductory guide to the Varian VNMR NMR processing / acquisition software. It is by no means intended to be all encompassing, but instead is designed to introduce the new NMR user to some of the most basic VNMR software features. With that in mind, this guide assumes that the user has little or no NMR experience. Some may feel that this guide doesn't go into enough detail. For those individuals, we have placed hyperlinks throughout the document in an effort to glean more details for those interested. For those interested, a large amount of additional NMR related information can be found on our website at <http://web.mit.edu/specplab/www>.

Conventions used in this guide are as follows:

- **Boldface** type indicates commands that are typed into the VNMR input window or in a terminal window
- Italic [*Boldface*] type with square brackets indicates a button in the VNMR menu that is to be pushed.
- <**Boldface**> type surrounded by a bracket indicate keyboard strokes.
- **LMB** indicates the Left Mouse Button
- **MMB** indicates the Middle Mouse Button
- **RMB** indicates the Right Mouse Button
- Unless otherwise noted, all commands typed into the input window are followed by an <↵ Enter> keystroke.
- When a command, parameter, or macro is first introduced a full definition or description will immediately follow.

Before we get started, we must first introduce some of the general features of the VNMR software interface. Upon initially opening the program (done by **single** clicking the VNMR application icon in the Common Desktop Environment (CDE) Toolbar shown at the right), the VNMR software display screen is divided into four distinct windows. These windows are called:

- 1.) The Input Window
- 2.) The Graphics Window
- 3.) The Text Window and
- 4.) The Acquisition Status Window.

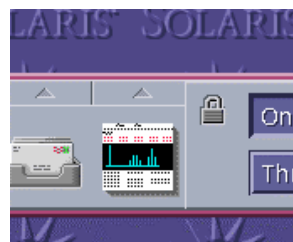


Figure 1: Small section of the CDE toolbar showing the VNMR icon

The significance of each of these windows will be discussed.

1. The Input Window:

The Input Window is typically located at the top of the display and is sub-divided into three subsections. A graphics representation can be seen in figure 2 below. VNMR system messages will be displayed in the Status Window (topmost portion), commands and parameters are typically input into the Input Window (middlemost portion), and menu selections can be made through the Menu Button Window (bottommost portion of the window).

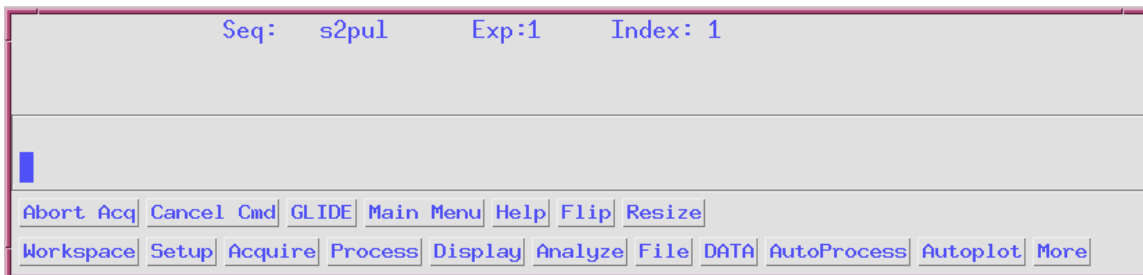


Figure 2: The VNMR Input Window. This is divided into the Status Window (Top), the Input Window (Middle), and the Menu Button Window (Bottom).

Most of the user input in this tutorial will be made through the Input Window and the Menu Button Window. Other points to note are that the pulse sequence (Seq: s2pul; a standard 2 pulse sequence), the experiment number (Exp:1), and the index number (Index:1) are displayed in the Status Window. This information will become more useful later.

2. The Graphics Window:

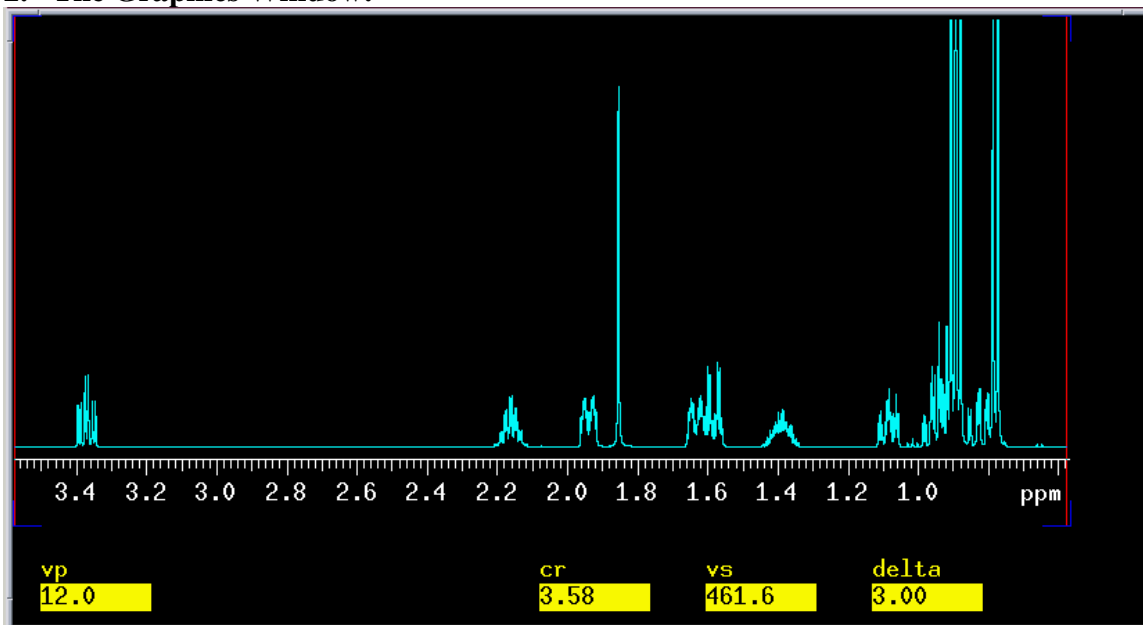


Figure 3: The VNMR Graphics Window

Here the user can graphically process visualize the newly acquired data. As an example we have shown here a small portion of a simple 1D ^1H spectrum of menthol ($\text{C}_{10}\text{H}_{20}\text{O}$) in deuterated chloroform (CDCl_3). Included in the window is the reference scale, vertical position (vp in mm) with respect to the bottom of the display, cursor position (cr in ppm), vertical scale (vs arbitrary units), and distance between the cursors (delta in ppm). This window can be thought of as a WYSIWYG (what you see is what you get) type of interface. Under normal circumstances, most plots are created just as they are seen in the Graphics Window. Here the user can process, zoom, expand, compare and so on the data they've acquired.

3. The Text Window:

The Text Window can have many different faces. Here text messages, manual pages, and certain analysis output can be displayed.

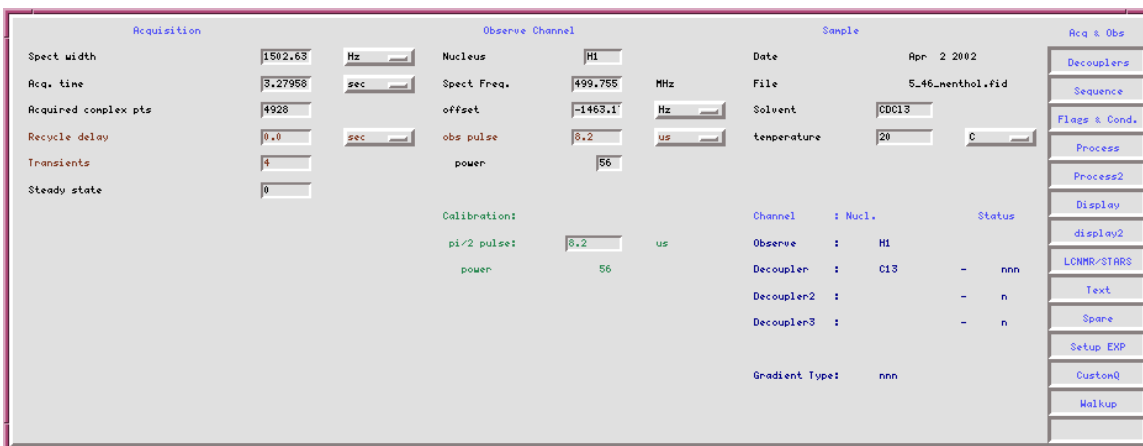


Figure 4: The Text Window. Note the buttons on the right-hand side. These allow for the easy visualization and changing of various parameters.

For example, the macro **man('bc')** (display the online description of command or macro ('bc', where **bc** (1 or 2D **baseline correction**) is the argument. This will display the manual pages for bc in the text window. Similarly, typing **dll** (**display listed line frequencies and intensities**) into the Input Window will display the peak index, frequency (in ppm), and the line intensity in the text window. Typing **dpir** will **display the peak integral regions**. A nice feature of the Text Window is the Tcl (call a "tickle") interface. The Tcl interface is shown in figure 4. This interface allows the user to simply highlight the desired parameter and enter a new value. This method can be used to change numerous acquisition parameters such as the **spectral width (sw)**, the **acquisition time (at)**, the **number of transients (nt)**, and so forth.

4. The Acquisition Status Window:

The VNMR software also has a small window called the Acquisition Status window, which is only available, when the workstation is connected to a spectrometer. The

Acquisition Status window is shown in Figure 5 and will be discussed in more detail shortly.

Getting Started:

Log onto the spectrometer workstation “mrhat” with your assigned reservation ID. This ID will be given to you by one of the DCIF staff members. If you do not have a user ID, you are not allowed access to any of the DCIF instrumentation. No exceptions. To get a user ID please see a DCIF staff member.

Open the VNMR program by either clicking on the VNMR icon in the CDE toolbar (Figure 1) or click the **RMB** on the desktop background and select VNMR (with the **LMB**) from the pull down menu.

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, then reopen VNMR.

You will be asked if you would like to load a shim file. Typically, you should load this file. At any later time you can also type **bestshim** to load the most recent ‘best shim’ values. These shim values are only intended as a reasonable starting place. The implications of the bestshim macro will be discussed in the ‘Shimming the sample’ section below. The macro is equivalent to typing **rts(‘/vnmr/shims/best’) su**. The command **rts** (retrieve shimfile) when fed an argument reads the argument (in this case the file /vnmr/shims/best) and **su** (setup) (followed by a beep) verifies the that the shimfile has been read to the console.

Occasionally the **su** command within the bestshim macro does not work. If “setup complete” is not displayed in the Status Window (see Figure 2), or you do not hear the computer beep, it didn't work. If this happens, type reset in the VNMR window to establish communication with the console. Or you may open a UNIX terminal window (see Figure 5). To do this, click the **RMB** on the desktop, and select **programs > terminal** or using the VNMR menu buttons select **[Main Menu] [More] [UNIX]**.

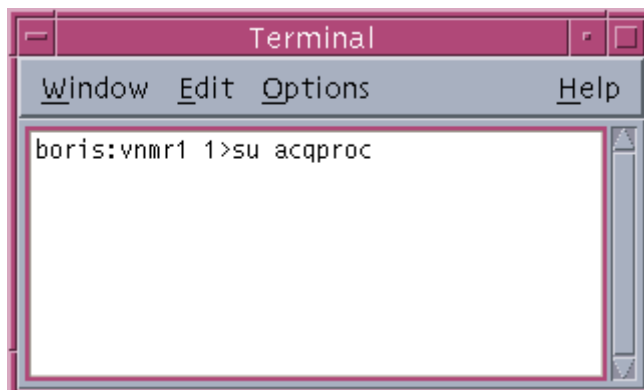


Figure 5: UNIX terminal window.

In the “terminal window” type **su acqproc** (this is the only macro that will not be defined) wait for the UNIX prompt to return then type **su acqproc** once again. The first **su acqproc** will kill the corrupt acquisition daemon (Status: will go from Idle to Inactive) and the second **su acqproc** will restart a fresh acquisition daemon (Status: will go from Inactive to Idle). The macro **reset** will also automatically stop and restart the acquisition daemon. See the following Figure for details.

reset is the equivalent of running two **su acqproc** command

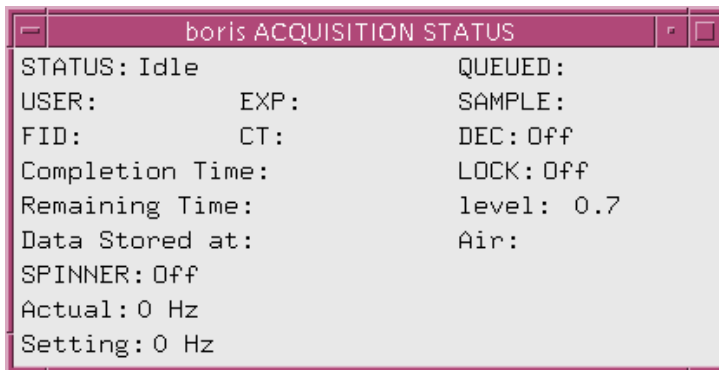


Figure 6: The Acquisition Status window. Note that the status here is 'idle'.

The login script will automatically change to your directory. To change to your directory manually: click [**Main Menu**], click [**File**], *select (highlight) your directory*, click [**Set Directory**]. These buttons may also be used to move around the directory tree.

Click [**main menu**], [**setup**] and choose the desired nucleus and solvent. The most common nuclei and solvents are available in ‘button’ form (i.e. [**C13,CDCl3**] indicates a ¹³C standard parameter set with deuterated chloroform as the solvent. If your solvent (or nucleus for that matter) is not listed, click [**other**] and input the desired nucleus / solvent. If, for example, you are interested in observing aluminum, when queried for **Enter Nucleus of Interest:** type **Al27**. If, on the other hand, if your solvent is deuterated ethanol, when queried for **Enter Solvent:** type any of the following: **Ethanol, Ethanol-d6, ethanol,** or **ethanol-d6**. Any of these permutations would work. For specific solvent details (shift, multiplicity, melting point, boiling point etc.), see the NMR Solvent Data Chart located next to or nearby the instrument. When finished, type **su** and wait for “setup complete” to appear in the Status Window. If it doesn’t, then see above instructions for **reset** or **su acqproc**. Once you’ve selected your nucleus and solvent don’t forget to type **su**! Forgetting to do so will on occasion make the instrument ‘un-tunable’.

Inserting the sample:

Click [*acqi*] in the Menu Button window to connect to the acquisition interface. The Interactive Acquisition window will open.

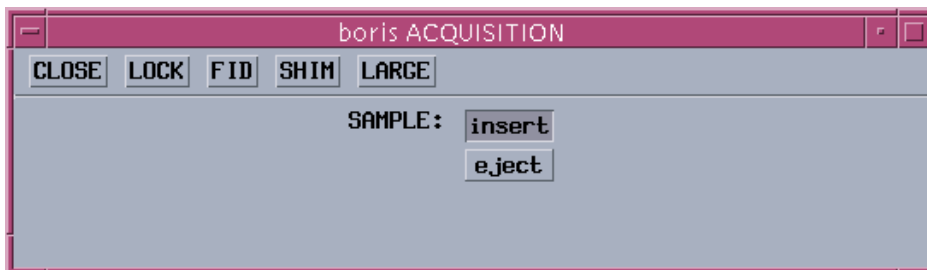


Figure 7: The Interactive Acquisition Window.

When the Interactive Acquisition window first opens, it should look just like the above example. If it does not (i.e. buttons are missing or the buttons are all 'grayed out'), you will need to do an **su acqproc** as described above.

Click [*lock*] and make sure that the spin is set to zero or turned [*off*] and lock is turned [*off*]. A recessed button indicates the command or parameter is off. A graphic representation is shown below with spin set to 20 but turned off and the lock turned off.

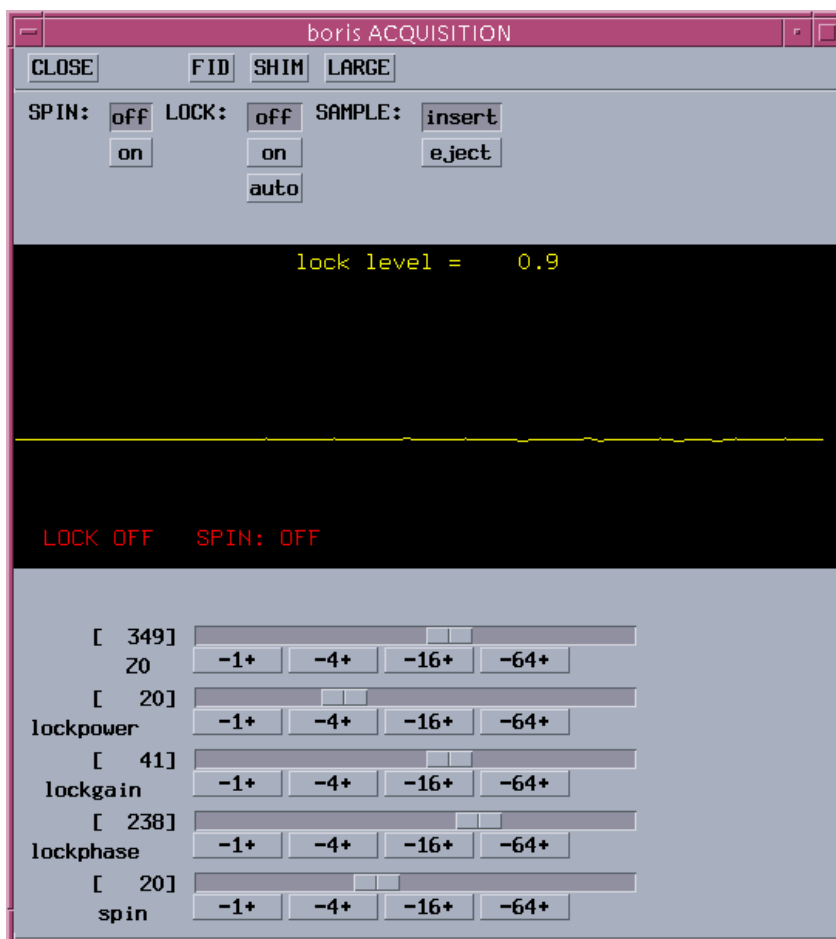


Figure 8: The 'Lock' window within the acquisition interface.

Note:

If the spin is [*on*], the air pressure may be too low and the sample may fall too quickly and break.

The adjustable parameters here are Z0 (magnetic field offset), lock power, lock gain, lock phase, and spin. Each of these may be adjusted by using either the slider bar (very coarse) or stepwise in increments of [± 1], [± 4], [± 16], or [± 64]. Details of each will be discussed shortly.

Clean the sample tube to remove any oils from handling. Kimwipes[®] are available at each of the NMR workstations. If not, more Kimwipes[®] can be found in the tan metal cabinet near the Bruker 600.

Proper sample volume and positioning is essential. Position the sample in the spinner using the gold colored depth gauge located nearby. Position the sample tube such that the center of the sample volume is at the center-line of the probe coil. Ideally, you would like to have 0.7ml total volume for a 5mm tube. The DCIF staff recommends using Wimad 528-PP or equivalent. These tubes are available in the VWR stockroom located n

the basement of Building 56. Under no circumstances should you use less than 0.5ml unless you are planning a variable temperature (VT) experiment. Improper sample volumes create problems in the local homogeneity of the field and will (at best) be more difficult to shim and produce inferior NMR data. Don't place the sample below the line marked, *PROBE BOTTOM*. Sample preparation tips and recommendations can be found [here](#)¹. A sample concentration calculator can be found [here](#)².

Clean the spinner and sample tube again (to remove oils from previous users).

Click [*eject*], wait for airflow, then place the sample at magnet bore opening.

CAUTION! Never place a sample in the magnet bore without air flowing. If you do, the sample will quickly fall and probably break. If that happens, call a staff member immediately—leaving it or trying to get it out yourself may cause damage to the probe and your reputation.

Click [*insert*], wait for a “click” sound (if you don't hear the click, then the sample is most likely not positioned correctly).

Click spin [*on*], or use the slider bar to set the spin rate to 20 Hz. Spinning the sample improves the effective magnetic field homogeneity for the bulk of the spins in the sample. Narrower line widths result. If the sample spin is not stable or does not spin at all, remove the sample, clean both the sample and spinner with a fresh Kimwipes[®] brand delicate task wiper, reinsert the sample, and turn the spinner back on. If the sample still doesn't spin contact a staff member for assistance. More help for you spinning woes may be found [here](#)³.

Locking the sample:

1. Optimizing the lock signal.

The magnetic field (as well as the frequencies generated by the oscillator) will drift and shift during data collection. A good experiment must compensate for these instabilities and this is why we lock. The “lock” may be considered a separate referencing experiment (in this case, deuterium). This deuterium experiment serves as a feedback loop to repetitively compensate for the magnetic field drift to keep the field constant (and keep good line resolution).

To lock your sample on the Mercury 300:

1. Be sure the lock is clicked [*off*].
2. Turn the *lock gain* to the maximum setting.
3. Turn the *lock power* to 30 or less.

The actual setting depends on your solvent, sample and the instrument.

These values have been determined sufficient for the Mercury. You should still check the system for lock saturation regardless.

- CDCl_3 (chloroform) use a lock power of $26 (\pm 2)$
- D_2O (water) use a lock power of $18 (\pm 1)$
- CD_3OD (methanol) use a lock power of $14 (\pm 4)$
- $\text{C}_2\text{D}_6\text{O}$ (acetone) use a lock power of $14 (\pm 4)$

4. Adjust the *Z0* slider until the frequency of the sine wave becomes zero (looks like a step function or a square wave) then turn the lock power down to 24 or less. Since this may seem unclear, a step by step graphical tutorial on how to lock can be found [here](#)⁴. If you are having lock difficulties, you can find additional help [here](#)⁵. Carefully adjust the *Z0* (using the [-I+] adjustment) until the maximum *lock level* is obtained. The *lock gain* may need to be decreased to find the optimum *Z0* setting. After *Z0* is optimized, turn the lock *[on]*. Suffice it to say it should look something like this:

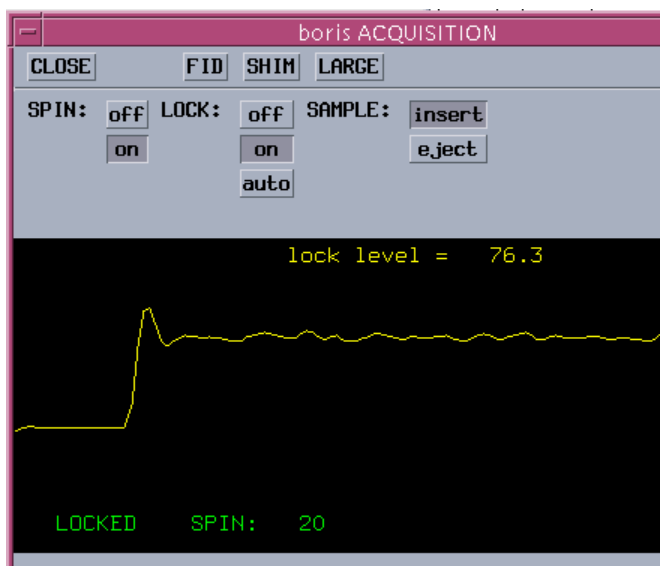


Figure 9: Sample locked.

Once the *Z0* field is optimized adjust the *lock gain* such that the lock level is at about 50%.

5. After the lock is clicked *[on]* adjust the *lock phase* to achieve the maximum *lock level*. The [-4+] adjustment should be sufficient. The current lock level can be seen at the top and center of the Interactive Acquisition graphics window (see Figure 9 above). Note that large changes to the *shims* affect the *lock phase* and this may need to be readjusted later.

6. If you are preparing for 2D-NMR or are using an unfamiliar solvent, it may be crucial to optimize the lock power. If the lock power is too high, the lock level will jump erratically (give the appearance of breathing). Excessive *lock power* produces lock saturation. The goal is to use the highest lock power you can without inducing saturation.

If the lock is not saturated, a linear correlation between the lock power and lock level is implied. To check this, decrease the *lock power* by 6 dB (in essence halving the power) and see if the *lock level* drops in half--if it doesn't drop in half, the sample was saturated at the previous lock power setting. CDCl_3 uses a *lock power* of 26 on the Mercury. Other solvents will require lower lock power settings (see above).

Shimming the sample:

Each sample perturbs the magnetic field differently. Shim adjustments are required to change the currents in 'corrective' coils surrounding the sample. These coils then perturb the magnetic field in such a way to (hopefully) make the field more homogeneous, producing better spectra. These coils are wrapped in such a way that they each affect the magnetic field *reasonably* independently. Unfortunately, physical constraints introduce "impurities" where the shim wires are physically positioned close together and consequently are influenced by the currents flowing through neighboring coils. Recall the importance of positioning the sample in the spinner. Correct and consistent volumes in the NMR tubes will be easier to shim.

Shimming can be performed by observing the lock level (higher \equiv more homogeneous) or shimming on the FID or spectrum. For now we will concentrate on shimming to increase the lock level. The lock level can be thought of as the *height* of the deuterium peak on which you are locked. The more homogeneous the magnetic field, the narrower the peak becomes. Since the area under the peak is constant, the peak height and lock level increase, as the field becomes more homogeneous.

Actually there are many shims that have already been adjusted for you when you type **bestshim** (assuming your sample volume, tube quality, etc. are correct). Most of the shims should not be adjusted for typical samples. If you are spinning, the shims with x- and y-axis components must not be touched; only the z-axis shims should be adjusted. How many of the z-axis shims you should adjust depends on your specific needs. Everyone should probably touch up the Z1C (course Z1) and Z2C (course Z2) shims. For some synthetic chemists with non-crowded spectra, this may be enough to see what is needed.

If the spectrum is crowded or if the line shape is important, Z1 (Z1 fine) and Z2 (Z2 fine) should be adjusted very carefully. Then if the spectrum indicates that more shimming is needed, the higher-order Z-axis shims may need to be carefully adjusted in a systematic fashion (vide infra).

Directions for shimming:

From the Interactive Acquisition window press [*shim*]

The Interactive Acquisition window will now be displayed in 'shim mode'. See the figure below. The window displays two bar graphs labeled 'coarse' and 'fine'.

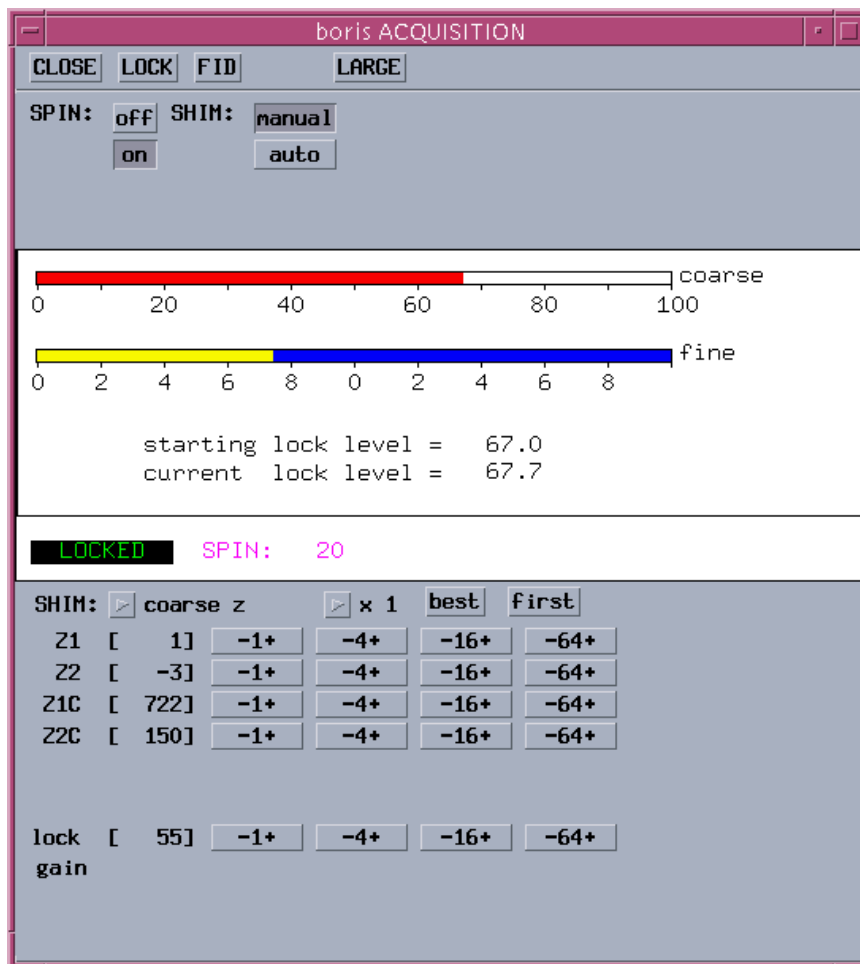


Figure 10: The 'shim' window of the Interactive Acquisition window.

Close inspection shows that the 'fine' scale is nothing more than an expansion of the twenty-unit range where the current lock level resides. The name of the game now is to adjust the shims in order to maximize the lock level.

At this point, we should probably discuss the macro **bestshim**. The bestshim macro does nothing more than retrieve a shimfile called 'best' and send a 'submit experiment' command (`rt('/vnmnr/shims/best') su`). This reads a 'reasonable' set of shim values to the console so the operator can spend a minimal amount of time shimming. However, many different things can affect how well (or poorly) the magnet shims. These contributing factors include the solvent, the solvent volume, the tube, and the tube placement in the spinner, the sample concentration, external perturbations, and others. Since we have found that well over 90% of our users use deuterated chloroform as their solvent, we create the bestshim file using 1% chloroform in chloroform-d₃, a Wilmad 528 tube and a total sample volume of 700µl. As we said, the solvent can have a large influence on shimming. If for example you are having difficulties shimming and are using D₂O as your solvent, once you've shimmed the magnet sufficiently well you may wish to save your own 'water shimfile'. To do this, type `svs` (save shimfile) and type a name for the file when prompted. Alternatively, typing `svs('my_water_shims')` will

save the current shim values to a file called 'my_water_shims'. To retrieve these values and read them to the console, rather than using the bestshim file type **rts('my_water_shims')** **su**. We try to keep the bestshim file as up to date as possible, but if the file seems to be deteriorating please notify a staff member.

Let's start shimming:

Start by clicking the [$\pm I$] button of Z1C (coarse) until the lock level is maximized. The lock gain may need to be decreased (any time the lock level approaches 100, decrease the lock gain until the lock level is ~70).

Next, click the [$\pm I$] button of Z2C (coarse) until the lock level is maximized.

As these two shims are coupled, iterate back and forth between Z1 (fine) and Z2 (fine) using the [± 4] or [$\pm I$] button, until a global maximum is achieved.

If the lock level is very noisy or unstable, the lock phase may need to be adjusted again (see step 5 in the section entitled "Locking the sample" above).

If needed, shim Z3 (then re-optimize Z1/Z2), then shim Z4 (then re-optimize Z1/Z2), and Z5 (then re-optimize Z1/Z2). Each time Z1 and Z2 should be interactively optimized to achieve the best magnetic homogeneity. Careful! Changing too many parameters at a time is counterproductive. If you get to a point where you only seem to be able to make the lock level worse, either click [*first*] (see Figure 10) or re-type **bestshim**. [*first*] will recall the shim values you initially started with when you first opened the Interactive Acquisition shim window and **bestshim** will simply allow you to start over. If you feel you've arrived at a point of no return, simply type **bestshim**. If you are still having troubles shimming go [here](#)⁶. If you would simply like more information about NMR spectroscopy and shimming check out Acorn NMR. They have a great five-part story about all the things you ever wanted to know about shimming but were afraid to ask! Check it out [here](#)⁷! Acorn NMR also has the "Shimming Ain't Magic" tutorial available for download in pdf format [here](#)⁸. These people do a great job and a wealth of useful information can be found on their website at <http://www.acornnmr.com>.

Typically only if running 2-D spectra will you need to turn the spin off and adjust the x-y shims. If you are seeing spinning side bands (often seen as small satellite peaks offset from a main peak by the spinning frequency), please contact a staff member. If you feel you need to adjust the x-y shims, please see a staff member for assistance.

After shimming, adjust the lock gain until the lock level is between 50 and 70.

Click [*close*] in the Interactive Acquisition window.

DO NOT TUNE THE MERCURY 300!

The Mercury 300 will only observe ^1H , ^{19}F , ^{13}C and ^{31}P . The selection of the nuclei is by software only for proton and fluorine. For carbon and phosphorus, you must also ensure that the proper quarter-wavelength cable is in place. Each cable is clearly labeled for ^{13}C or ^{31}P . Ask a staff member if you are unsure how to connect the appropriate cable.

You are now ready to acquire data!

Some normal (or default) settings and commands:

- **gain='n'** (the gain is 'not used') This will allow the instrument to automatically adjust the receiver gain. If you get an autogain failure message in the Status Window, decrease the pulse width by typing **pw=pw/2 ga**. (pulsewidth=pulsewidth / 2)
- **nt** is the number of transients (or scans). The default is 16 for proton acquisition. Increase (decrease) for dilute (concentrated) samples by typing **nt=64** (or **nt=4**)
- **dg** will display the parameters in the Text Window.
- **dps** will display the pulse sequence being used in the Graphics Window. A simple "presaturation" pulse sequence is shown below.

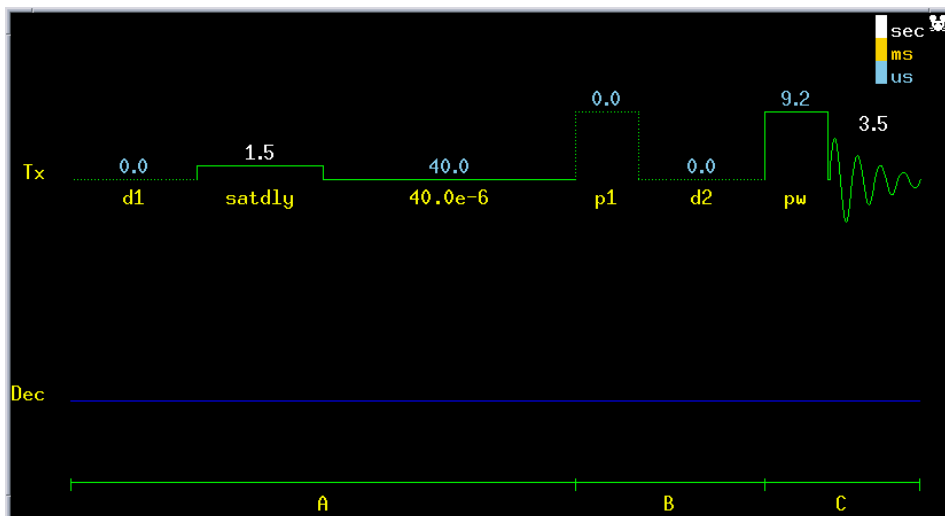


Figure 11: A simple pulse sequence.

Type **ga** (submit the experiment to acquisition and FT the result) to start collecting the spectrum.

If **nt** is large (>128), **bs** (the **block size**) should be used (set to say 16) to ensure the collection is going well. This tells the acquisition computer to stop every '**bs**' scans long enough to send data over to the user computer so that the user can begin to process the data before the experiment finishes. The data will continue to be collected until the

experiment finishes. Type **wft** (weight, Fourier transform) after you see the message, “BS 1 completed”. Each time **wft** is typed, all the data collected thus far are used for the weighted Fourier transform.

At any time, you can stop the acquisition (saving the number of scans done at last **bs**) by typing **sa** (**s**top **a**cquisition after next transient is collected) or **aa** (**a**bort **a**cquisition, now!).

After the data are collected, the fid needs to be processed...

ffav is a macro which does the following 5 commands

- f** (set display parameters to **full** spectrum)
- full** (set display limits for a **full** screen)
- aph** (automatic **p**hasing of rp and lp)
- vsadj** (automatic **v**ertical **s**cale **a**djustment)
- dscale** (**d**isplay **s**cale below spectrum or FID)

You can click the [*phase*] button to phase manually. Click with the left mouse button (0 order phasing) near the right edge of the spectrum and drag up and down to see the effect. Try the right mouse button for fine phase adjustment (1st order phasing). Next click farther to the right in the spectrum, and phase until the baseline is flat. If you really mess it up, type **lp=0 rp=0 ds** (sets the left and right phase to zero and displays the spectrum) and start over. With practice (and proper expansion) you should be able to do a better job phasing the peaks of interest by hand than with **aph**.

An expansion of a selected region may be done as follows. Select a region in the Graphics Window by defining the region with the **LMB** and then the **RMB**, then click [*expand*]. Click on the spectrum near, say, the chloroform peak then type **nl** (nearest line).

To reference the spectrum, click the spectrum with the LMB. Then type **nl** (nearest line) and **rl(7.27p)** (reference line) for chloroform or whatever shift is appropriate for that resonance (see NMR Solvent Data Chart for a list of solvent reference peaks).

Type **dscale** to display the entire **scale**.

Type **ds** to displays spectrum (refreshes screen).

Save the file: (Files are not automatically saved.)

All users will have a directory for saving files. This directory is named after your user ID and is created when you first login. All of your data should be saved in this or subdirectories of this ‘main’ directory. If you would like to create a subdirectory: Click [*Main Menu*], click [*File*], then type **mkdir(‘Xxx’)** where Xxx is the new subdirectory name. Click [*File*] again to update the display. Select (highlight) the newly created directory and click [*Set Directory*]. All files will now be saved here. Type **svfz** save your data to a file. The **svfz** command will also save a copy of your data set to the workstation 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 the spectrometer only. The command **rt** will allow you to retrieve a file. You should already be in your home directory, but if not, type **gohome**.

jexp1 will allow you to join experiment #1. **jexp2** will allow you to join experiment #2, allowing you to load data or parameters in one experiment while processing data in another. So for example, one can process and plot the proton spectrum while a carbon experiment is being acquired in another experiment. **explib** displays the list of available experiments. New experiments can be created by typing **cexp(#)**. Typing **man('jexp')** will give more information.

A list of some of the more common commands and parameters can be found [here](#)⁹.

Many people choose to do the rest of the processing and printing on zippy (which is free). However, all of the processing works on mrhat as well.

What to do when you are finished:

Click [**acqi**] and then [**lock**]. Turn spin [**off**] and turn lock [**off**]. Then [**eject**] the sample, remove it from the top of the magnet, and click [**insert**] to turn off eject air.

Type **exit** to close the VNMR software.

Then log out (right click, select [**log out**], or select EXIT from the CDE menu bar).

Sign the book. Report any problems, no matter how small.

To work on zippy:

Login to zippy using X-Win32.

Open the VNMR software as is done on the spectrometer

Find the file saved on mrhat:

Click [**Main Menu**]

Click [**File**]

Highlight the *mrhat* directory, click [**Set Directory**]

Highlight the *filename* of interest, click [**Load**]

Type **wft** to weight, Fourier transform

Integrate the spectrum:

- Click [**partial integral**] to display the integral line. Type **cz** (clear zeros from memory), then click [**resets**]. Use the **LMB** to define those regions you wish to integrate. The **RMB** can be used to undo mistakes. Use the **MMB** to adjust the **is** (integral scale). To assign an integral value to a particular region, type **ds** to regain cursor control then use the **LMB** to 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 display the integral values under the spectrum.

Other useful integral related commands / macros:

isadj will automatically adjust the integral scale.

io integral offset (with respect to the spectrum).

dc will perform a drift correction on the spectrum.

bc will do a baseline correction. Will give more ‘accurate’ integral values.

Add some text to be printed with the spectrum:

Type **text**(‘your name here’). This will print *your name here* on the spectrum. You might also want to try the macro **gettext**. This will open a small easy-to-use text editor, which will easily allow you to place text on multiple lines.

Print out some stuff:

Type **pl pscale pir pap page**

Will print the spectrum on the screen at that time, as well as the scale, the integral resets and the acquisition parameters (which will include the text you added). Page sends to the printer. The command line **pl pscale pir pap** is plotted (with the command, page) to the printer whereas the line, **ds dscale dpir dg** is the print-to-screen equivalent.

Try this:

Type **dpf**

Adjust the threshold if necessary on the *[Th]* button (may need to click the *[Interactive]* button). Type **dll**.

For more information about plotting a line list, for example, type **man(‘pll’)**

Then try typing **dpfhz** (is macro that is: **axis=’h’ ppf axis=’p’**)

pl pscale pphz page

man(‘pl’) will display the manual page for the **pl** command.

A listing of the many of the most common plotting and printing commands can be found [here](#)¹⁰.

When you are finished on zippy:

Type **exit** to close the VNMR software.

Then log out (right click, select *[log out]*, or select EXIT from the CDE menu bar).

¹ <http://web.mit.edu/speclab/www/tips.htm#five> (Sample preparation tips.)

² <http://web.mit.edu/speclab/www/tips.htm#five> (Sample concentration calculator)

³ <http://web.mit.edu/speclab/www/tips.htm#four> (Why won't my sample spin?)

⁴ <http://web.mit.edu/speclab/www/tips.htm#four> (Graphical lock tutorial)

⁵ <http://web.mit.edu/speclab/www/tips.htm#four> (Something is wrong. I can't lock!)

⁶ <http://web.mit.edu/speclab/www/tips.htm#four> (My sample won't shim...)

⁷ http://www.acornnmr.com/nmr_topics.htm (Acorn NMR shimming info.)

⁸ <http://www.acornnmr.com/Downloads/shimming.pdf> (Shimming Ain't Magic download (.pdf))

⁹ <http://web.mit.edu/speclab/www/vnmrop.html> (some common commands)

¹⁰ <http://web.mit.edu/speclab/www/tips.htm#three> (plotting and printing tips)

Running Fluorine or Phosphorus NMR on the Varian 300

1. Login, load the **bestshim** file, and then load ^{19}F or ^{31}P parameters for your solvent. Click *Setup* and then *Nucleus, Solvent*. Select *F19* or *31P* and your solvent. Type **su**.
2. Insert your sample into the NMR as you normally would (Don't forget to turn on the eject air!).
3. Click *Acqi*, then the *Lock* tab and lock as you normally would. Adjust Z0 until you are on resonance. Note the value of Z0.
4. Click the *Shim* tab, and shim normally.
5. Click on the *Lock* tab again, and turn off the lock.
6. Eject your sample and insert the neat CFCl_3 (for ^{19}F) or H_3PO_4 (for ^{31}P) standard.
7. Do not change the Z0 value or attempt to shim on the standard.
8. Type **nt=1 gain=0**, then **ga** to acquire one scan.
9. Reference the peak to 0ppm. Left click on the peak, click *Ref*, and then type **0**.
10. Eject the standard and insert your sample again.
11. Do not change the Z0 value. You should still be on resonance.
12. Do NOT turn on the lock. In order to make sure that your resulting spectrum is still referenced correctly, you want to run unlocked.
13. Type **gain = 'n'**. This turns on the Autogain.
14. Change **nt** to however many scans you wish to acquire.
15. Acquire your spectrum and save it.

Note: If the instrument status (in the Acquisition Status box) shows that you are stuck in the *Auto Set Gain* step, type **aa** to stop the process. Set **d1=10 nt=1**, and type **ga** to acquire one scan. This should set an appropriate gain for your sample. Then type **gain='y'** and **nt=1e6 bs=16**. Type **ga** to start the acquisition. After one block size (16 scans) transform your FID (type **wft**) and take a look at your spectrum. If the signal-to-noise ratio is ok, then type **sa** and save your data.

Because you are running unlocked, it is not recommended you run for an extended period of time.

Presaturation for Solvent Suppression on the Mercury 300 NMR.

This applies only to the 300 MHz Varian NMR in the DCIF. Anything written in **bold type** is a direct command that is to be typed in the VNMR command prompt.

1. Insert your sample, lock, shim well, and load the correct proton parameters. Take a normal 1D ¹H spectrum.
2. You can choose a desired spectral width by setting the cursors and pressing the *Expand* button. Type **movesw**.
3. Type **dn='H1'**. This changes the decoupler nucleus to proton.
4. Type **PRESAT**.
5. Place the cursor on the peak you want to suppress. Type **sd**.
6. Make sure the following parameters are set as follows:
satfrq=dof
satmode='ynn'
satdly=1.5
satpwr=2
7. Make sure that **dm='nnn'**.
8. Set **nt=1** and **ss=4**. Take one scan. Verify that the 'target' peak has been suppressed.
9. If the peak has not been sufficiently suppressed, you can increase your **satpwr**. Never set your **satpwr** greater than 12 because that will cause damage to the probe.
10. Set **nt** to whatever value you want, acquire spectrum, and save it.