Magnetic Resonance Center

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OPERATING INSTRUCTIONS FOR VARIAN NMR SPECTROMETERS RUNNING VNMRJ

Sections: Page#

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1. Introduction:

The NMR center has served more than 2000 research scholars in the past years, and currently serves ~100 users. This current JAVA based version of VNMRJ offers user-friendly graphical user interface (GUI) along with automated approaches to quickly acquire a good quality spectrum

This handout is intended to serve as a guide to understand this new version of the software without much ado. In fact, for experienced users the shift would be nearly effortless as the easiness of usage is more in the new version. The default procedure of being checked out still hold good, i.e. one has to book an appointment with Dr. Dinesh Sukumaran when she/he feels comfortable with operating the spectrometer. Until one gets checked out, he/she ***must*** be accompanied by a trained-checked out researcher while practicing using the instrument. This material is essential, however isn't the sole requirement, for being checked out.

Suggestion to improve this handout are encouraged, please email them to the NMR GA's email.

2. Sign-up

Go to online webpage at http://www.nmrcenter.buffalo.edu/.

3. Sample preparation:

Typically ~700-750µL of sample volume is desired. In order to get a reasonable 1D spectrum within a few minutes, about 5-10mg and 20-50mg of solid/liquid sample is required for ^{1}H and ^{13}C NMR, respectively. In case you are working with other nuclei (such as ^{19}F, ^{31}P, etc.) that are as sensitive as ^{1}H the sample concentrations requirements are similar to that of proton.

Choose an appropriate deuterated solvent for your sample depending on its solubility and also the temperature you would like to work in, i.e. for an experiment that involves recording spectra at sub-zero temperatures water (D_2O) is not a suitable candidate as it freezes and for experiments involving temperatures higher than 50°C chloroform ($CDCl_3$) is not fit as it boils around 61°C. Also the solvent used must have deuterium ($^{2}H \equiv D$) in it; the necessity of the same would be discussed in the LOCK section. Make sure the solvent you are choosing doesn't interfere with your peaks of interest. That is, if your desired peaks are in the aromatic region it might be wise to avoid using solvents with aromatic moieties (e.g. benzene-d6, pyridine-d5). Common solvents used in NMR are: heavy water (D_2O), chloroform ($CDCl_3$), dimethyl-sulfoxide-d6 (DMSO, C_2D_6SO), trifluoroacetic acid ($C_2F_3O_2D$), methanol-d4 (CD_3OD), acetonitrile-d3 (C_2D_3N), pyridine-d5 (C_5ND_5), benzene-d6 (C_6D_6).

Additional points:

(a) Make sure there are no solid/insoluble particles floating around in the solution. These would make shimming difficult. In the case that you are using automated shimming methods, such samples would not be shimmed properly and might worsen the already

existing shim-sets. In such cases, it might be prudent to filter the sample before loading it on to the NMR tubes.

(b) Though higher the sample concentration the better, after certain concentration samples tend to be become viscous, in which case once again the shimming gets difficult. So its not always the more the merrier!

(c) It is always a good practice to have ~700-750uL of sample which makes shimming a lot easier. This volume in a 5mm regular wilmad NMR tube translates to about ~6.5-7.5cm.

4. Logging in, creating and working with directories, opening VNMRJ:

In the welcome screen, log-in with your username (generally your P.I.'s last name) and password (collect it from your lab). It's a good idea to have a directory for each user in every lab, so that its easy to retrieve and also make regular back-up of your data time to time. Remember linux treats characters with case-sensitivity, i.e. the folder with name Nmr is different from the nmr and NMR.

Useful commands:

Throughout this document you would find a set of useful commands that might come handy while working with the spectrometer. Remember that the syntax that VNMRJ uses is a little different from what the linux terminal uses. You would find both listed in below, enter the Varian commands in VNMRJ and Linux commands in a terminal/console/konsole.

Varian command –

mkdir('foldername')	– creates a folder with name "foldername"
cd('foldername')	– changes directory to "foldername" (if it exists)
cd	– changes directory to your home directory
pwd	– print working directory (the one you are currently in)

Linux command –

> mkdir foldername	– creates a folder with name "foldername"
> cd foldername	– changes directory to "foldername" (if it exists)
> cd	– changes directory to your home directory
> pwd	– print working directory (the one you are currently in)

Once logged in, by default the user will be in his/her home directory. Let's take an example username as "vnmr1". The home directory would be /home/vnmr1/. From your home, you could create/delete directories within VNMRJ/linux-terminal with the commands as listed above.

An example using a linux terminal is used for illustration, similar commands produce similar outputs in VNMRJ window.

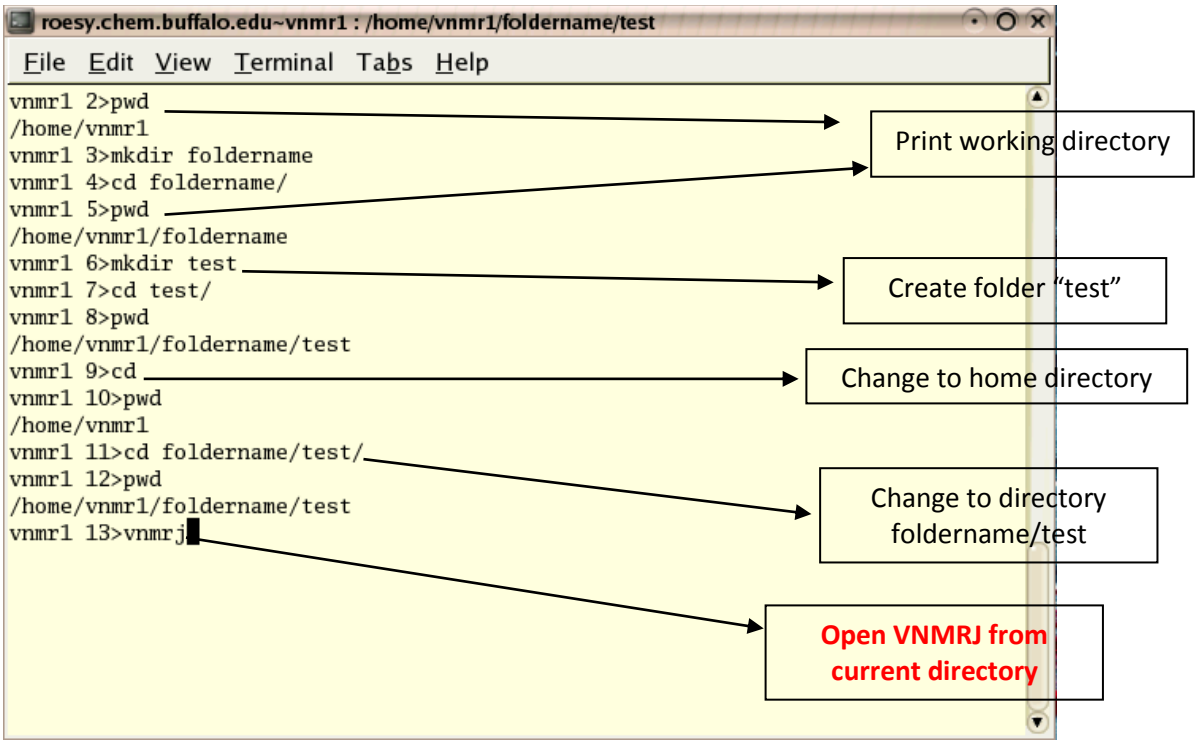


Figure 1: Linux terminal – the command line interface.

Start the vnmrJ program (used to control NMR Spectrometer) by double clicking the VNMRJ icon on your desktop.

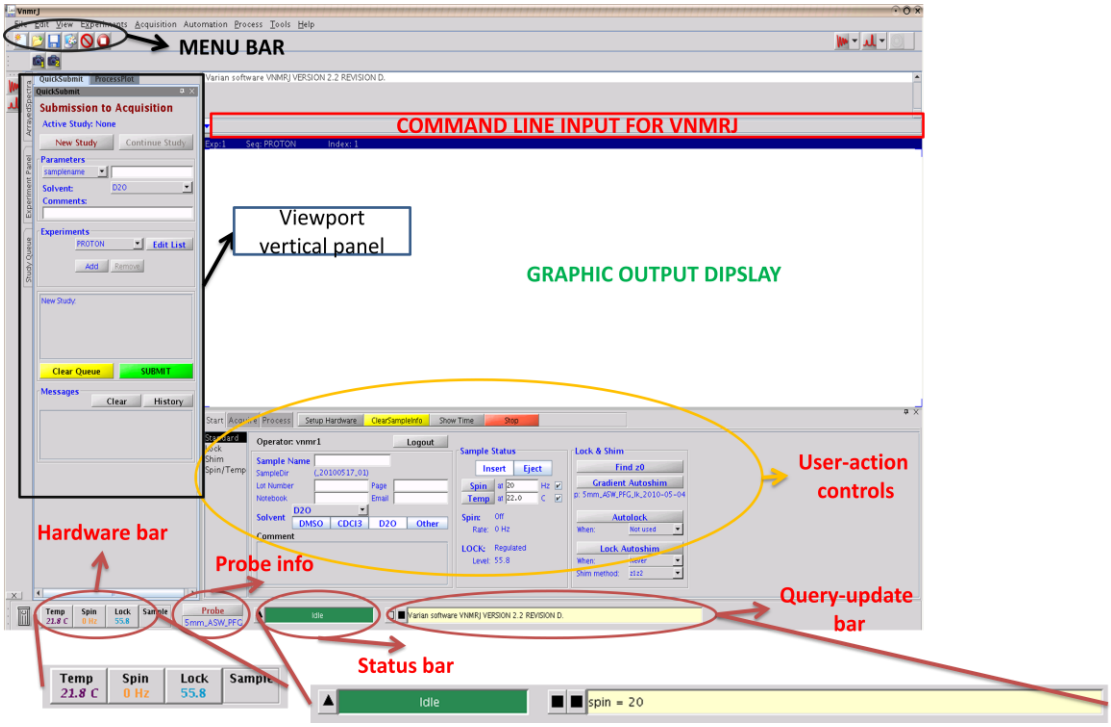


Figure 2a: VNMRJ user interface.

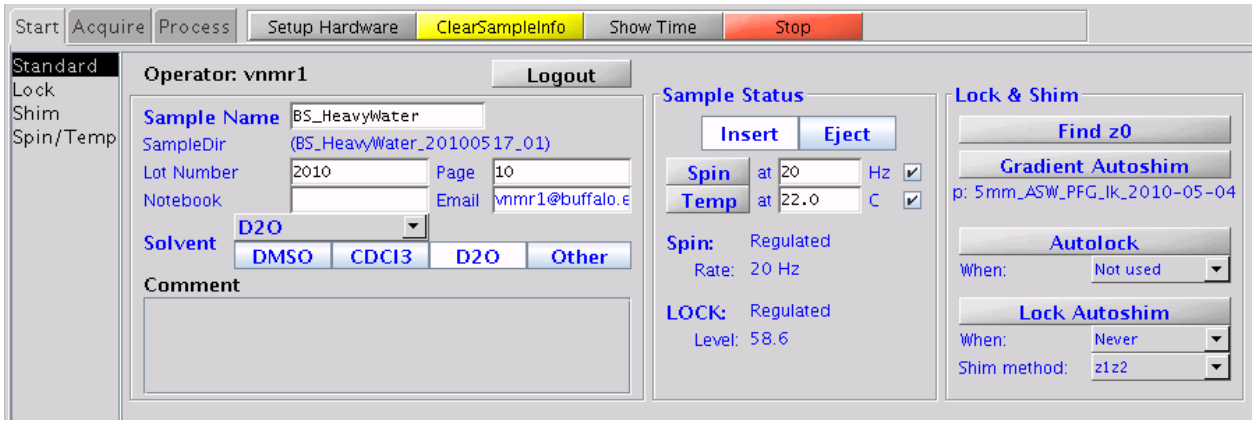


Figure 2b: User-action controls panel.

5. Loading the sample

The spectrometer by default must always be locked with the reference sample (D_2O lock-standard sample). First unlock the spectrometer from the present sample, START>LOCK>Lock "OFF" (figure 4b.). Then eject the already present sample by clicking "Eject" in the "Sample Status" portion of the user-action controls panel (see figure 2b; START>STANDARD>SAMPLE STATUS). The sample tube should be positioned properly in spinner to make sure that the transmitter-receiver coil is able to *see* the sample. This is calibrated by using a sample depth gauge each time before inserting the sample. Place the sample with care in the spinner and by using the gauge place the sample right at the center of the dotted rectangle shown in the bottom of the gauge, with equal volume of solution above and below the rectangle. The default gauge setting is at **67**; NEVER change this setting. In figure 3 (below) this is set to 70 and the dotted rectangle can be found between the readings 35-51.

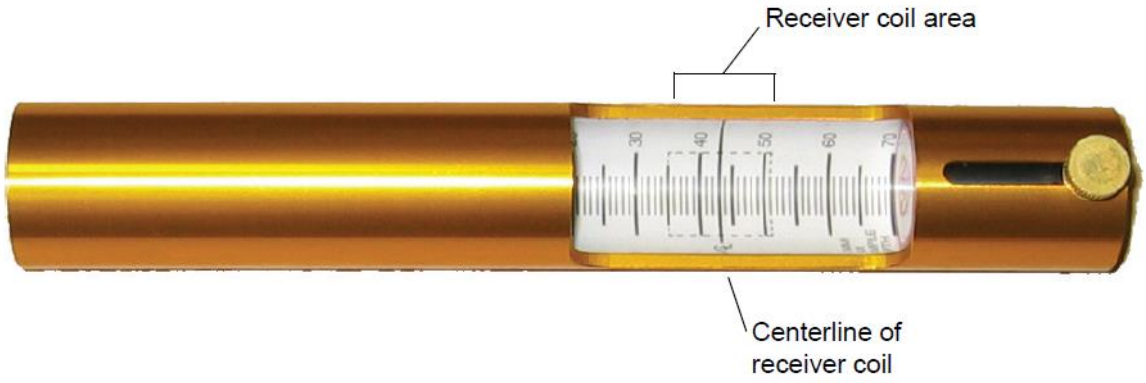


Figure 3: Sample depth gauge.

Once well placed, gently load the sample in the spectrometer and click "Insert" in the "Sample Status" panel (see figure 4b) to insert the sample.

6. Locking the sample:

Modern day NMR spectrometers comprise of gigantic high field super conducting electromagnets whose magnetic field strength drifts (~10Hz/hr) with time due to several reasons. In order to correct for the same, the deuterium signal from the solvent is made use of, the deuterium frequency of the lock solvent is referenced to the ^{2}H resonance frequency of the spectrometer and maintained a constant throughout the experiment. That is, when the magnetic field drifts a current opposing such a change is compensated by the lock circuitry to maintain the same lock level. Thus, it becomes important to always keep the spectrometer locked over time, as a large drift might be very difficult to tackle after a point. So, remember to leave the spectrometer locked with the some sample, preferably the lock-standard sample.

The sample is locked by activating the "Lock" section of the user-actions control panel (START>LOCK, see figure 4b)

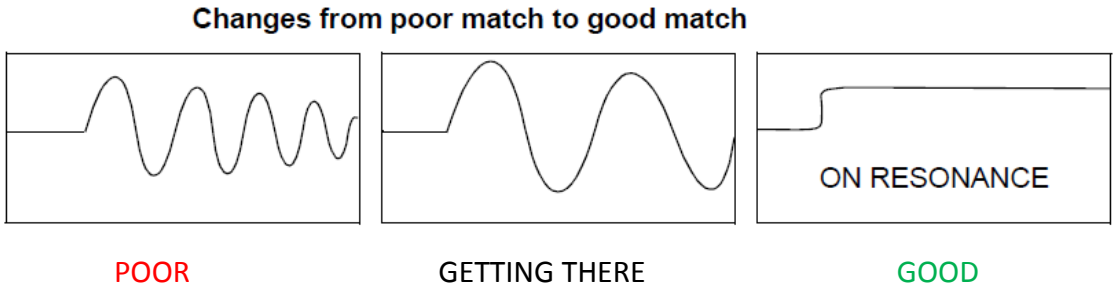


Figure 4a: Approaching the lock.

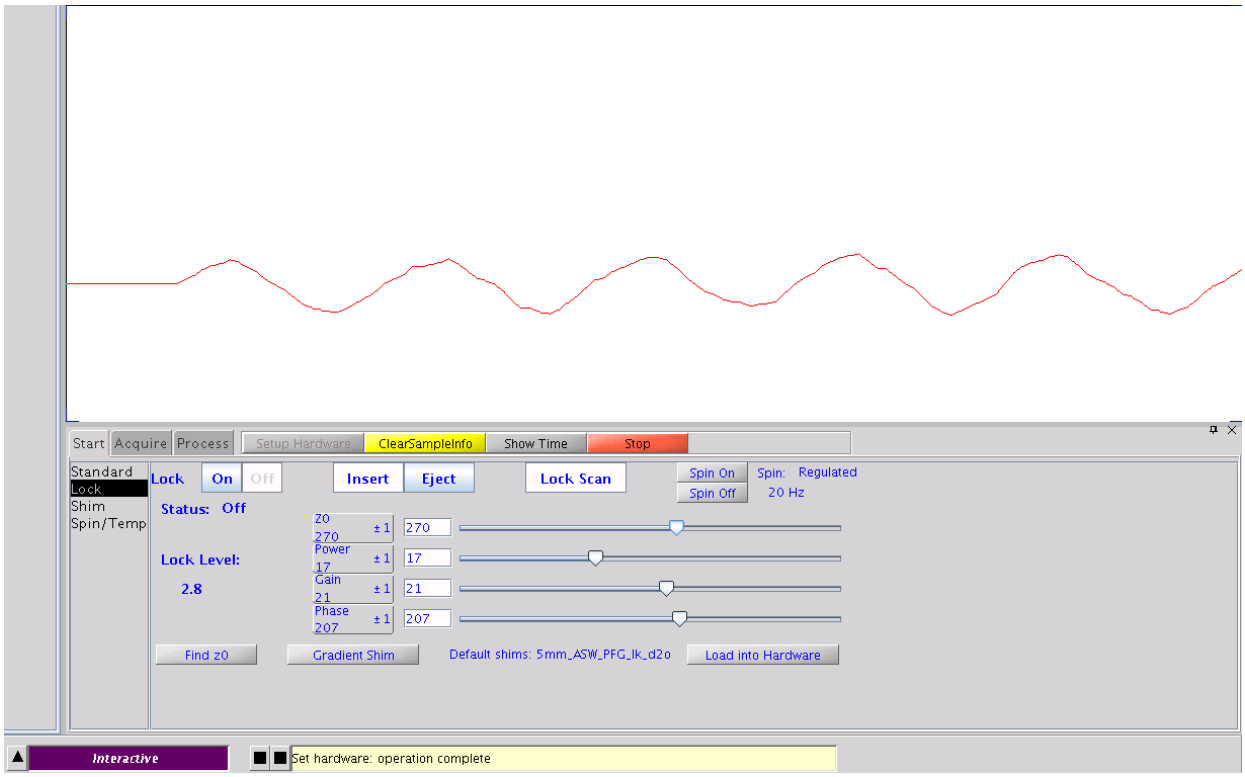


Figure 4b: Lock panel – the lock scan shows the typical pattern when "Z0" is off.

Figure 4c: Lock panel – after the sample is locked.

Locking can be imagined to be a procedure where the two frequencies (one from sample and another from spectrometer) are subtracted from each other. In the case when the frequency difference is high, one typically observes a wavy patter as shown in figure 3b., this implies that the "Z0" is off. In order to lock the sample, first turn on the "LOCK SCAN" and observe the wavy pattern that appears. Once the LOCK SCAN is turned on, one would observe that the spectrometer status changes from *idle* to *interactive.* Alter the "Z0" value until the "number of waves" reduces to null and a step function appears (figure 3c.). The best approach would be first go in one direction (either increase OR decrease) the "Z0" value, and observe whether the number of waves increases or decreases, in the case that it increases then the direction you are taking is opposite and therefore reverse the direction, vice-versa, and you would start to see the number of waves reducing.

Once you have reached a good lock level, click "LOCK ON" and turn off "LOCK SCAN". And adjust the lock phase to check whether the lock level increases further. If yes, iterate between "Z0" (lock "off") and lock-phase (lock "on") to maximize the lock level. A lock level of ~40-50 units (in the above example it is 69 units) is good enough. After locking if you are higher/lower than this level then decrease/increase the lock power-lock gain accordingly so that you are around this value. It is always a good practice to maintain lock power lower than the lock gain (ratio of power:gain ~ 1:1.5).

A lower power would ensure that the sample isn't "saturated" with irradiation, in which case the shimming process might get misled. To check whether the sample is being saturated, increase the lock power by 6 units and see whether the lock level nearly doubles. For example, if at a lock power of 16 units the lock level is 40 units, then increase power to 22 units and check whether the lock level reaches around 80 units. If yes, then the sample is not getting saturated; reduce the lower power back to 16 and continue with your experiment. If not, work around lock power-gain to establish a condition where the sample isn't getting saturated and at the same time maintains a good lock level. This shouldn't be a big issue with the organic samples with completely deuterated solvents.

7. Mouse clicks

Most steps in setting up the experiment involves manipulating a string of variables (e.g. the "Z0" for lock, "Z1" and "Z2" for shims, etc.), which is done by using mouse clicks on the respective radio button (i.e. Z0 for "Z0" and "Z2" for Z2). Left/right click is used to increase/decrease the variable by a given increment, and the middle click helps in toggling between the various possible increments (1, 5/10, 100).

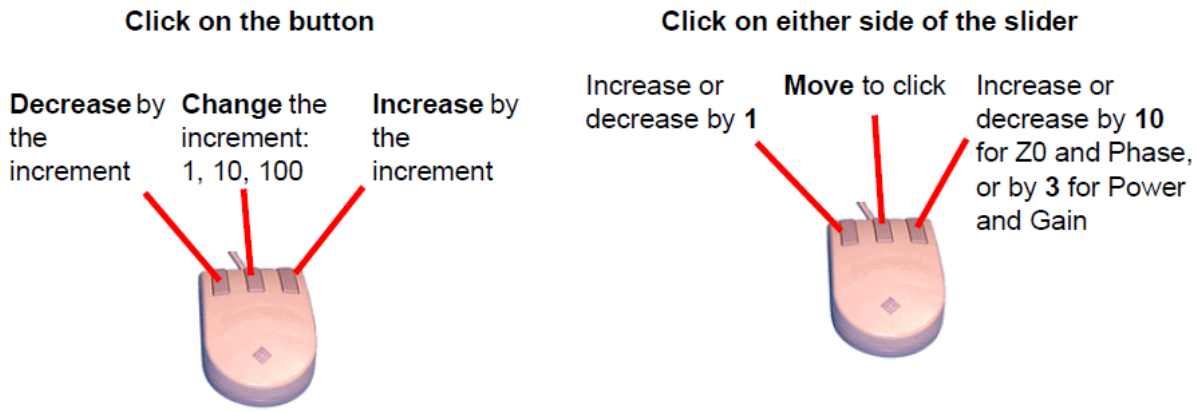


Figure 5: Mouse clicks and their functions.

8. Shimming:

The most important condition to satisfy in order acquire a good NMR spectrum is to ensure a homogenous magnetic field around the sample. The external magnetic field provided by the super-conducting magnetic requires fine tuning in order to further improve the homogeneity. This is done by altering currents passing through the electromagnetic coils (shims coils – Z1, Z2 … Z5, X, Y, XZ, YZ, XY, etc.). This involves manipulating variables (largely "Z1", "Z2") to observe an increase in the lock level (similar to the procedure adopted while locking by altering "Z0"). Shimming is straight forward for most samples, however might be demanding for challenging samples. Scientific judgment and patience are pertinent to drive a shimming attempt to better results.

The follow flowchart might serve as a basic guide for locking and shimming.

a) Turn lock off, eject previous sample out
b) Position your sample using the sample depth gauge
c) Insert your sample
d) LOCK
    i. Adjust "Z0" to lock the sample and to attain the maximum possible lock level with the given settings of lock-power and lock-gain. Remember to adjust "Z0" only when lock is OFF.
    ii. Turn lock ON
    iii. Adjust lock-phase to maximize the lock level
    iv. Iterate steps i, ii, and iii until no further increase is seen with iteration steps
e) SHIMMING
    i. Adjust "Z1" to maximize the lock level
    ii. Adjust "Z2" to maximize the lock level
    iii. Iterate the above two steps until no further
f) Check a quick spectrum ^{1}H spectrum

i. Experiment set-up: Select the experiment you would like to perform from the Menu bar (Experiments > Proton)
ii. Parameters for a quick experiment: ***nt = 1, ss = 0, d1 = 0, gain = 26*** (nt – number of transients, steady state scans, recycle delay, receiver gain, respectively)
iii. Start acquisition: ***ga***
iv. Process and check spectrum: ***ft cdc dc*** (Fourier Transform, cancel drift correction, apply drift correction, to the signal respectively and display the spectrum)
v. Expand a reference peak (depending on the solvent) to check for the shims*
TMS peak ~0 ppm – look for the singlet shape
DMSO peak ~2.5 ppm – look for quintet multiplicity
$CDCl_3$ peak ~7.3 ppm – look for the singlet shape

g) If you are happy with the spectrum, increase the number of transients, steady state scans and recycle delay to desired value depending on the signal to noise observed(e.g. ***nt = 16, ss = 2 d1 = 2***).

* - shims can be evaluated by checking how "narrow" the lines are in the spectrum. Click the cursor on the reference peak and type ***nl*** to place the cursor on the nearest maximum intensity resonance and type ***res*** to check the resolution. A half line-width (50% line width) of ~1-1.5Hz implies a moderately shimmed spectrum, lower the half line-width the better.

The shim window in VNMRJ looks as shown below:

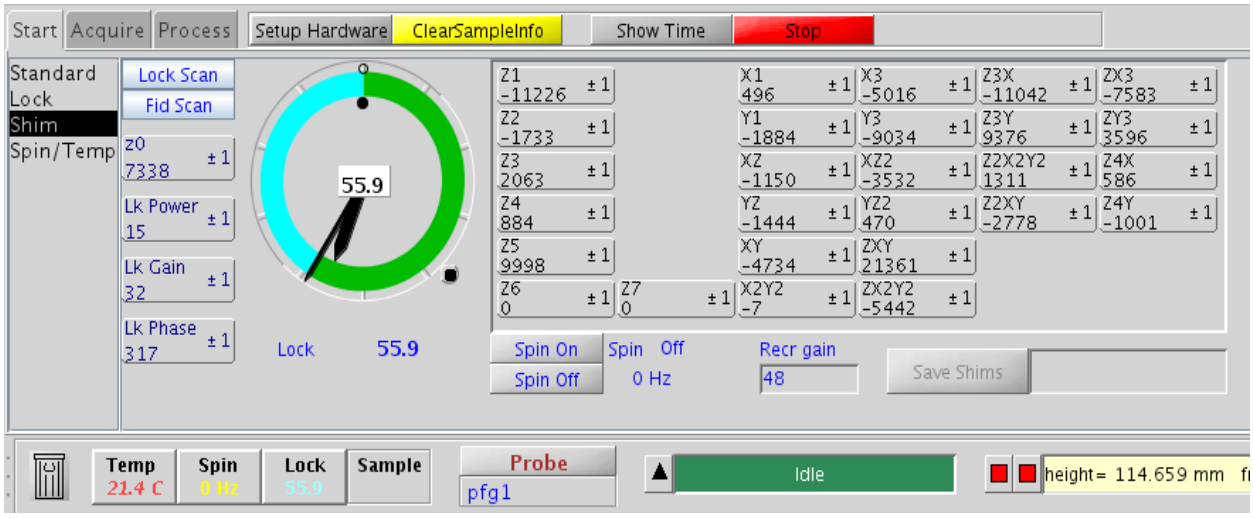


Figure 6: Shimming panel

The above procedure involves shimming only the Z1-Z2 shims while certain occasions might call for more rigorous shimming involving the other shim coils. However, students are NOT ENCOURAGED to alter these shims without having a sound knowledge of the science behind it. An easy way to check whether non-spin shims are OK, is to set the spin OFF and check the drop in lock level. If it drops around 5-8 units, then non-spin shims are OK, however if it drops more than 15 units then it implies that the non-spin shims are off. The default shim-set can be recalled by typing ***sos su*** which will retrieve back the previously saved good shim-set to the

current setting. It is advised to touch up the lock and shims after retrieving the parameters, and one might have to set up the experiment again.

9. Automatic locking and shimming:

Locking and shimming can be done automatically for a quick set up. However user supervision and judgment is required to make sure the procedure has worked fine. The sample can be auto locked by clicking the "Find Z0" found in the START>STANDARD>LOCK & SHIM menu. It is always a good practice to check for the optimal Z0 manually after auto-locking. Gradient auto shimming can be done after the sample has been locked (START>STANDARD>LOCK & SHIM → Gradient Autoshim). It is wise to save the beginning shims sets (***svs('filename')***) before auto shimming, which would come in rescue if gradient shimming didn't deliver for that instant.

10. Experiment set-up:

Spinning the sample helps in improving shimming, thus it is recommended to spin the sample at 20 Hz for all 1D experiments. However, due to certain experimental requirements it is better not to spin the sample for multidimensional experiments. The spin setting can be adjusted in the "Sample Status" panel (START>STANDARD window, see figure 2b).

The sample temperature can also be varied according to sample/experiment requirements. To reach temperatures higher than room temperature no further arrangements are required and the desired temperature can be attained by just changing the temperature value in the "Sample Status" panel. However to reach temperatures lower than room temperature, the cooler unit (if present) requires to be activated. NOTE: Please make sure the solvent you are working with doesn't undergo phase transitions while altering the temperature (i.e. should not freeze/boil). In either case the chances of accidents/acquiring damage to the spectrometer probe is very likely. Always consult with your lab members/NMR GA/P.I. before venturing into such endeavors.

This version of VNMRJ has been set up in a fashion where an arsenal of one-dimensional and multidimensional NMR experiments can be set-up with ease. The entire list of experiments can be found from the "Experiments" drop down menu and also in the "Viewport vertical panel" (see figure 2b). The set up is intuitive to set up almost any experiment under the sun with this.

Once the experiment has been set up, type ***go*** or ***ga*** <u>once</u> to begin the data acquisition. One would observe that the spectrometer status changes from "Idle" to experiment time count down once data acquisition begins. After the data acquisition is completed, the data can be processed for analysis.

11. Processing and important parameters:

Since 1970 NMR has used routinely Fourier Transform mode of data acquisition. In this mode, the sample is excited by a short burst of high energy radiofrequency pulse and the response of

the system is recorded as the time domain response. As time and frequency are inverse Fourier pairs, Fourier Transformation of the time domain response yields the desired frequency domain spectrum. However, the data can be processed in simple ways to extract information of interest. Thus, data processing forms an integral part of NMR data acquisition. The following commands are be used for data processing, by no means this is an exhaustive list of commands. For entire list please refer to the VNMRJ manual. The commands are listed here in no particular order.

Experimental parameters: (numbers in brackets indicate suggested values)

(a) nt – Number of Transients (16)
(b) ss – Steady-state Scans (dummy scans) (2)
(c) d1 – recycle/relaxation delay between successive scans (2)
(d) at – Acquisition time (2)
(e) gain – Receiver Gain (26)
(f) ga – submit experiment to acquisition and FT the result
(g) go – submit experiment to acquisition
(h) aa – Abort Acquisition
(i) temp – TEMPerature (‘n’)
(j) spin – SPIN value (in Hz) (20)
(k) sa – Stop Acquisition
(l) ra – Resume Acquisition
(m) bs – Block Size (8)
(n) sw – Spectral Width (15 ppm)
(o) tof – Transmitter OFfset
(p) np – total Number of Points (sum of real and imaginary points)
(q) pw – Pulse Width
(r) pw90 – 90° pulse width duration
(s) tn – Transmitter Nucleus
(t) dn – Decoupler Nucleus
(u) dof – Decoupler OFfset
(v) ni – Number of Increments (2D parameter)
(w) sw1 – spectral width of indirect dimension
(x) array – set up an array to be performed (e.g. for pw calibration, tof adjustment, etc.)
(y) dm – Decoupler Mode for first decoupler (set with dn)
(z) dmm – Decoupler Modulation Mode for first decoupler

Processing parameters:

(a) lb – Line Broadening (in Hz, by default is an exponential function)
(b) fn – Number of points for Fourier Transformation after zero padding (generally 2 times np)

(c) rp – right phase (in degrees, zero order phase)
(d) lp – left phase (in degrees, first order phase)
(e) rl – reference line (syntax ***rl(2.5p)*** sets the referred peak to 2.5 ppm)
(f) ph – PHase sensitive mode
(g) av – Absolute Value mode
(h) ft – Fourier Transform
(i) wft – Windowed Fourier Transform (ft command including line broadening and zero padding)
(j) aph0 – Automatic Zero Order Phase Correction
(k) aph – Automatic Phase Correction
(l) cdc – Cancel Drift Correction
(m) dc – Drift Correct
(n) bc – Baseline Correction (after the desired integrals have been set)
(o) cz – Clear integrals
(p) isadj – Integral Scale ADJust (after the desired integrals have been set)
(q) vsadj – Vertical Scale ADJust

Display parameters:

(a) cr – Cursor position in the direct dimension
(b) ds – Display Spectrum
(c) df – Display FID
(d) dg – Display Group of acquisition/processing parameters (you must be in the text window to view it)
(e) delta – cursor difference between the left and right cursors in direct dimension
(f) dps – Display Pulse Sequence
(g) nl – Nearest Line (sets the cursor to the nearest maximum peak)
(h) res – RESolution of the most intense peak in the displayed spectrum
(a) dpcon – Display CONtour (syntax ***dpcon(14,1.4)*** display on screen with 14 contour lines with 1.4 spacing between them)
(b) dconi – Interactive 2D data display
(c) dll - Display Listed Line frequencies and intensities
(d) f - Set display parameters to full spectrum
(e) full - Set display limits for a full screen
(f) wc – Width of Chart (wc2 for indirect dimension)
(g) sc – Start of Chart (sc2 for indirect dimension)
(h) wp – Width of the Plot in direct dimension (wp1 for indirect dimension)

Plotting parameters:

(i) pl – PLot
(j) ppa – Print PAramaters
(k) pap – Print All Parameters
(l) pscale – Print ppm SCALE

(m) pir – Print IntegRals
(n) page – plot to a PAGE
(o) dpirn – Display and Print IntegRals Normalized
(p) ppf – Plot Peak Frequencies over spectrum (set th)
(q) th – THreshold
(r) linelist – print resonance frequencies for lines above threshold
(s) intlist – print integral list (part/full integrals must be turned on)
(t) pps – Plot Pulse Sequence
(u) pcon – Plot CONtour (syntax ***pcon(14,1.4)*** with 14 contour lines with 1.4 spacing between them)

Miscellaneous commands:

(a) svf – SaVe FID (syntax ***svf('filename')*** saves FID as 'filename.fid' in the current directory)
(b) rt – ReTrieve FID (syntax ***rt('filename')*** reads the FID 'filename.fid' from the current directory)
(c) rtp – ReTrieve Parameters from FID (syntax ***rtp('filename')*** retrieves parameters from the FID 'filename.fid' present in the current directory)
(d) svs – SaVe Shimset (syntax ***svs('shimname')*** saves current shims as shimname in default shims directory)
(e) rts – ReTrieve Shimset (syntax ***rts('shimname') load='y' su*** reads current shims as shimname from default shims directory and sets it up)
(f) mf – Move FID (syntax ***mf(1,2)*** moves FID from experiment 1 to experiment 2)
(g) mp – Move Parameters (syntax ***mp(1,2)*** moves parameters from experiment 1 to experiment 2)
(h) cexp – Create EXPeriment (syntax ***cexp(1)*** creates new experiment 1)
(i) jexp – Join EXPeriment (syntax ***jexp(2)*** joins experiment 2)
(j) r1 – r7 – Real-value storage for macros
(k) n1, n2, n3 – Name storage for macros
(l) exit – exit VNMRJ

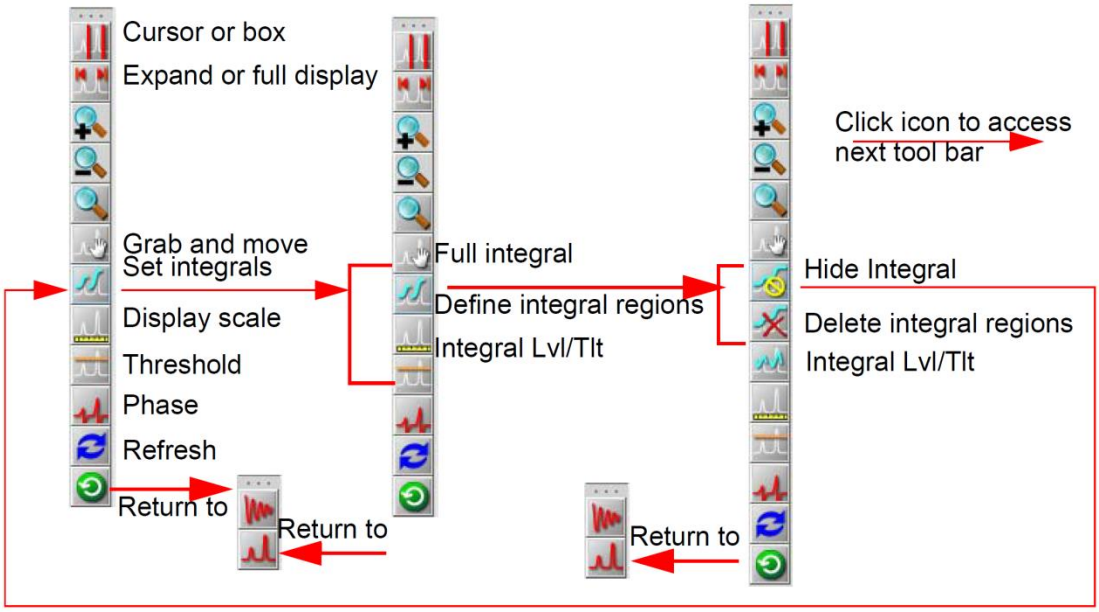
The GUI can also be used for quick and easy data processing and for spectrum/FID display. Here is the summary of the icons that and their functions (the following images have been taken from VNMRJ manuals):

Icon	*Description*
	Zoom in.
	Zoom out.
	Select zoom region.
	Redraw display.
	Return to previous tool menu.

1D Display Spectrum Toolbar Controls

Icon	*Description*
	One cursor in use, click to toggle to two cursors.
	Two cursors in use, click to toggle to one cursor.
	Click to expand to full spectral display.
	Pan or move spectral region.
	Display integral.
	Display scale.
	Toggle threshold on or off.
	Phase spectrum.

Integration



Display FID Toolbar Controls

Icon	*Description*
	One cursor in use, click to toggle to two cursor.
	Two cursors in use, click to toggle to one cursor.
	Click to expand to full FID display.
	Pan and stretch.
	Click to show real and imaginary.
	Click to show real and zero imaginary.
	Click to show real only.
	Toggle scale on and off.
	Phase FID.

Icon	*Description*
	One cursor in use, click to toggle to two cursors.
	Two cursors in use, click to toggle to one cursor.
	Click to expand to full display.
	Pan and stretch.
	Show trace.
	Show projections.
	Click on  to show horizontal maximum projection across the top of the 2D display.
	Click on  to show horizontal sum projection across the top of the 2D display.
	Click on  to show vertical maximum projection down the left side of the 2D display.
	Click on  to show vertical sum projection down the left side of the 2D display.
	Rotate axes.
	Increase vertical scale 20%.
	Decrease vertical scale 20%.
	Phase spectrum.

Interactive weighting can also be done by typing ***wti*** in the command line prompt. The mouse click can be used to alter various parameters for optimal data processing.

Check box	*Icon*	*Function*
Line broadening		Selects line broadening or exponential weighting. A negative value gives resolution enhancement.
Sinebell		Selects the sinebell constant. A negative value gives squared sinebell. Change sign by clicking outside the box at the left.
Shifted Sinebell		Selects the sinebell shift constant (if sinebell is active).
Gaussian		Selects the Gaussian time constant.
Shifted Gaussian		Selects the Gaussian shift constant (if Gaussian is active).
Additive weighting		Selects the additive weighting constant.
Return		Returns to the previous menu.

Figure 7: Interactive Weighting window for data processing

12. Logout

After completion of the experiments, make sure the data is saved in the pertinent directory. Then replace your sample with the standard D_2O lock sample and lock the spectrometer. In

case you have altered the temperature, set the temperature back to room temperature by typing ***temp='n' su***. Turn off the cooler unit if you had turned it on. Wait until the temperature reaches equilibrium. In case the "Lock Scan" tab is turned on, turn it off and then type ***exit*** in the VNMRJ command line prompt and then log out of the systems (Start Menu Bar > Actions > Logout). In case that when one logs out when LOCK SCAN is not turned off the spectrometer status gets hung in the *interactive* mode and cannot be used until it is reset. Please make ensure such mishaps do not occur.

13. Spectrometer Maintenance and NMR Center usage:

The cryo-magnet needs to be maintained regularly for sustained usage. Every week liquid Nitrogen (LN2) is topped off for all three spectrometers on Wednesdays between 4 – 5PM. Students are strongly discouraged from booking/using the spectrometers during this period. For every 6 – 7 weeks liquid Helium needs to be filled for all three spectrometers. This schedule, unlike LN2, is not fixed and is dependent on Helium boil-off rate and cryogen availability. The spectrometers would not be functional 2 hours before liquid Helium filling and 2 hours after filling. This is done to ensure that the magnet returns to equilibrium before usage.

Regularly on Wednesdays the NMR GA runs spectra for outside samples between 1 – 3 PM on the 500MHz, and if necessary also on the 400MHz. Apart from that, depending on inflow of such samples, experiment(s) might be run any time in the week and schedule can be seen from the online schedule sheet.

14. Regulations and protocol:

(a) ONLY persons who have been checked out by Dr. Dinesh Sukumaran may use the NMR/EPR spectrometer. Students can be trained only by lab members who have been checked out. In case one has to run spectra for samples and has not been checked out, he/she should get help from their lab members and run the spectra with their assistance.
(b) Report instrument failures and accidents (i.e. broken tube in the probe) as soon as they happen to the NMR GA/Dinesh. If neither is present, leave a note with the nature of the problem and what time the problem was observed. Instruments that are offline need to be repaired quickly for the benefit of everyone.
(c) Observe the sign-up rules and DO NOT EXPECT ANOTHER USER TO SURRENDER THEIR TIME because you are running late. When signing up for NMR time, do make sure you include your group's phone number. If you will not be using the time you signed up for, remember to cancel your time. Calling the next user to notify him/her that you are finished early is a nice courtesy.
(d) Do not attempt experiments that you have not been trained for. Once you are checked out, you still need to be initially supervised for low temperature experiments.
(e) In depth spectral analysis should be performed only using the NMR data station computers.
(f) Place used tissues, scrap paper and kimwipes in the garbage.

(g) Do not handle anything in the NMR room when wearing lab gloves.
(h) Do not bring metallic/magnetic objects near the magnet. This includes ATM/credit cards, watches, electronic devices (MP3 players, mobile phones, USB drives, etc), keys, etc.
(i) Always lock on the standard sample when done with the instrument and select standard 1D parameters. Type ***su <return>***.
(j) Malicious users may lose facility privileges.
(k) Back up your data routinely.

References:

(1) Derome. A.E., Modern NMR Techniques for Chemistry Research, 1st ed.; Pergamon: Oxford, c1987. UB library call code: QD 96 N8 D47 1987
(2) Rahman, Atta-ur-, One and Two Dimensional NMR Spectroscopy; Elsevier: 1989, chapter 1. UB library call code: QD 96 N8 D35 1989
(3) Varian Manuals:
    a. NMR Spectroscopy User Guide: VNMRJ 2.2D
    b. VNMR Liquids NMR User Guide: VNMRJ 2.2D
    c. VNMR User programming
    d. VNMR Pulse Sequences
    e. VNMR Command and Parameter reference
(4) Cavanagh, John, et. al., Protein NMR Spectroscopy; Academic Press: 2006, chapter 3. ISBN-13: 978-0121644918