

# **Agilent NMR User's Manual**

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## Suggested Reference Materials

### *Books*

High-Resolution NMR Techniques in Organic Chemistry by Claridge, Timothy D.W.

NMR Spectroscopy Explained by Jacobsen, N.E.

Structure Determination of Organic Compounds by Pretsch, E.; Bühlmann, P.; Affolter, C

200 and More NMR Experiments by Berger, S. and Braun S.

### *Articles*

Kwan, E.K., Huang, S.G. "Structural Elucidation with NMR Spectroscopy: Practical Strategies for Organic Chemists", *Eur. J. Org. Chem.* **2008**, 2671-2688

### *Web Resource*

Chem 605 Course Website at the University of Wisconsin-Madison

<http://www.chem.wisc.edu/areas/reich/chem605/index.htm>

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# I. Abbreviations and Terms

COSY	Correlation Spectroscopy
DQF-COSY	Double-quantum filtered correlation spectroscopy
f1	“Indirectly” detected dimension
f2	“Directly” detected dimension
FID	Free Induction Decay
HMBC	Heteronuclear Multiple Bond Coherence
HMQC	Heteronuclear Multiple Quantum Coherence
HSQC	Heteronuclear Single Quantum Coherence
Hz	Hertz
MHz	Megahertz
MW	Molecular Weight
NMR	Nuclear Magnetic Resonance
NOE	Nuclear Overhauser Effect
NOESY	Nuclear Overhauser Effect Spectroscopy
PFG	Pulsed field gradient
ppm	parts per million
probe	part of the spectrometer where RF signal is sent and received
Proton	used interchangeably for $^1\text{H}$
RF	radio frequency
ROESY	Rotating frame Overhauser effect spectroscopy
T1	Longitudinal Relaxation
T2	Transverse Relaxation
TOCSY	Total Correlation Spectroscopy
VNMRJ	Varian’s software used to interface with console
VT	variable temperature control
X-nucleus	used to refer to anything not $^1\text{H}$

## II. Common VnmrJ Commands

### Spectrometer Control Commands

cexp(#) – create new experiment. e.g. cexp(20) will create experiment 20  
dg – display group of acquisition/processing parameters  
dps – display pulse sequence  
e – eject sample  
i – insert sample  
jexp# – join/go to experiment #. e.g. jexp20 will join/go to experiment 20  
su – setup experiment. Force hardware to use current parameter settings

### Acquisition Parameters

aa – abort current running experiment  
at – acquisition time in seconds  
bs – block size of data periodically read and stored on disk  
dn – decoupler nucleus  
ga – submit current experiment and autoprocess the data when available  
gain – receiver gain  
go – submit current experiment  
movesw – move new spectral window to the area enclosed by box cursor  
nt – number of transients/scans  
ss – number of steady state transients  
time – display total experiment time with current parameters  
tn – observe nucleus

### Data Processing Commands

aph – automatic phasing of spectrum  
cz – clear integral resets  
dc – drift correction. Adjusts spectrum baseline so it is close to zero  
dpf – display peak frequencies  
dpir – display integral values  
dssa – display arrayed spectra in a stack plot  
dssh – display arrayed spectra horizontally  
dssl – label displayed arrayed spectra  
ff – display the full spectrum and maximize the window size  
full – display maximum window size  
lb – line broadening amount for exponential window function  
mf(n,m) – move FID file from experiment n to experiment m  
nl – move cursor to nearest line  
svf – save the data, you will be prompted to enter a file name  
wft- weighted Fourier transform. Transform the FID to spectrum with applied window function

## Display Parameters

axis='x' – axis label in display and plots. axis='p' gives ppm. axis='h' give hertz

cr – cursor position

delta – in two-cursor mode stores the width between the two cursor

dsx – UTSA macro. Shows the full spectrum fit to the graphic display window

vp – vertical position of spectrum, in mm

vs – vertical scale of spectrum

vsadj – automatic adjustment of spectrum so the highest peak fits window height

## Plotting Commands

**UTSA print macros** – these will autoplot spectra as described

print – print spectrum with peak frequencies

iprint – print spectrum with integrals

fprint – print spectrum with integrals and peak frequencies

## **Agilent Commands**

page – submit plot to printer

pap – plot all parameters in table format

pir – plot integrals

pl – plot spectrum

pll – plot frequency line list in table format

ppa – plot basic parameters

ppf – plot peak frequencies

pltext – plot user comments/notes

### III. NMR Instruments and Probes

The chemistry department has two NMR spectrometers and three workstations running VnmrJ 4.2 for offline processing in BSE 0.110.

#### The Spectrometers:

**Mercury Plus 300** – 300 MHz Mercury Plus console running VnmrJ 4.2 software. The Mercury Plus is a two channel instrument with a broadband H/F/X probe. This spectrometer is for acquiring routine  $^1\text{H}$ ,  $^2\text{H}$ ,  $^{13}\text{C}$ , and  $^{19}\text{F}$  spectra. If monitoring a reaction by NMR the Mercury Plus 300 is to be used.

**DD2 500** – 500 MHz DD2 console running VnmrJ 4.2 software. The DD2 is a two channel instrument with pulsed field gradients and shaped pulse capability for a wide variety of 1D and 2D experiments with the following probes. The DD2 500 should be used to collect publication quality spectra and to perform advanced 1D and 2D experiments as well as variable temperature experiments.

**ID (default probe)** – 5 mm probe with pulsed field gradients.  $^1\text{H}$  optimized. X-channel 50-202 MHz ( $^{15}\text{N}$ - $^{31}\text{P}$ ). VT – 20-60 °C. Best for sample limited samples and indirect detect experiments.

**SW** – 5 mm probe with pulsed field gradients. X-channel 50-203 MHz ( $^{15}\text{N}$ - $^{31}\text{P}$ ). VT – 20-90 °C

**10 mm BB** – 10 mm broad band probe. X-channel 50-202 MHz ( $^{15}\text{N}$ - $^{31}\text{P}$ ). VT – 20-90 °C.

If you would like to change the probe in the DD2 500, see Dr. Wherritt for assistance.

## IV. Instrument Reservations

All reservations for UTSA instruments is done with the chemistry department's Booked Scheduler (<https://booked.chemistry.utsa.edu>). Once training has been completed you will receive an account to reserve instrument time.

You MUST have a reservation to use the spectrometers!

### Scheduling Rules

You MUST have a reservation to use the spectrometers!

- 1) Reservations can be made up to 3 days in advance
- 2) Prime time is 8 AM to 7 PM on weekdays.
- 3) Blocks of time are 10 min in length and up to three blocks in a row (30 min) may be reserved during prime time
- 4) A total of 1 hour may be reserved during prime time in a day per user. e.g. Six 10 min reservations or two 30 min reservations
- 5) On weekends, users may reserve a total of 6 consecutive blocks (60 min) on a given instrument.
- 6) Reservations for the same time slots on multiple instruments is not allowed.
- 7) Users may only reserve two (2) overnight experiments during the week on a single instrument.
- 8) Using other user's accounts to schedule multiple blocks simultaneously is forbidden at all times!
- 9) You are responsible for using a time slot when you've reserved it. Always cancel reservation you will not use.

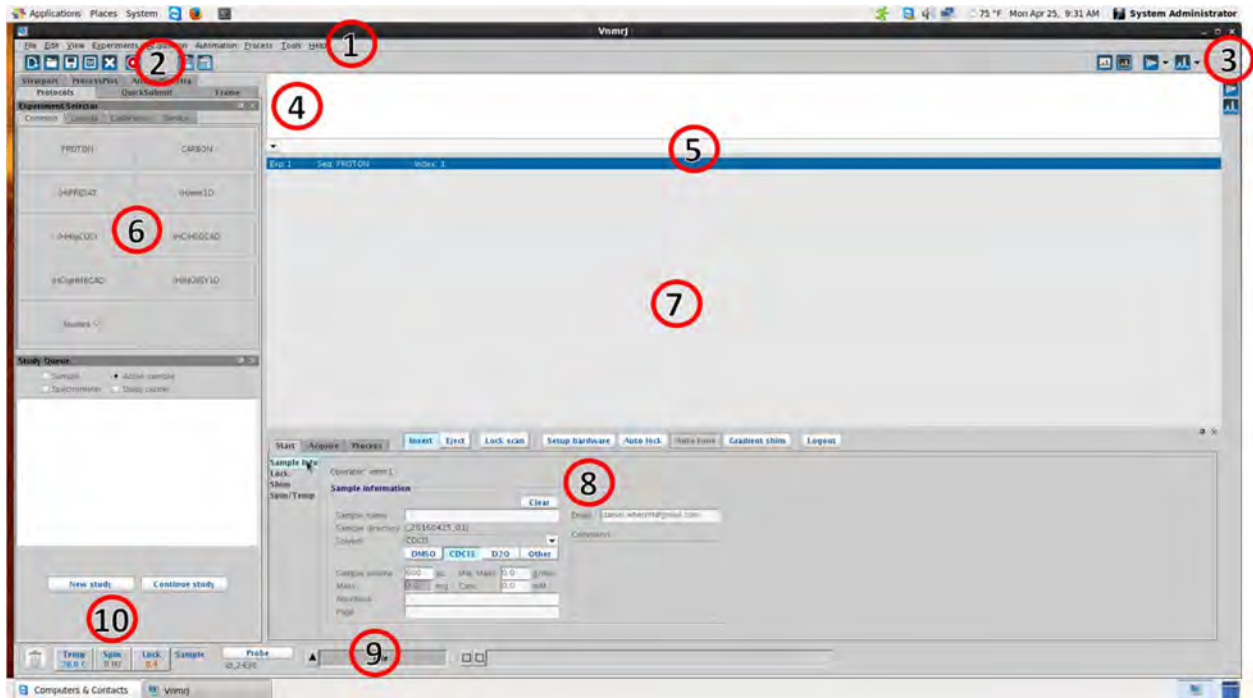
Failure to abide by these rules will result in the suspension of NMR privilege for one week. Repeated (>3) disregard for these rules will result in permanent loss of NMR privileges.



## V. Safety

- 1) Only faculty, staff, authorized users and trainees are allowed in the facility. Visitors are allowed with escorts. No social guests are permitted.
- 2) NMR spectrometers are large very powerful electromagnets. DO NOT bring metallic items inside of the 5 gauss line (black circle on the floor around the magnets) as they could get stuck to the magnet. Remove your keys, cell phone, and wallet from your pockets before entering the 5 gauss line. Failure to do so could result in your phone no longer working and your IDs and credit/debit cards being erased by the magnet.
- 3) Inside of the NMR magnets are liquid nitrogen and liquid helium to make the main magnet coil a super conductor. During normal operation these do not pose any threat to user. However, if the magnet should quench (lose its super conduction and start to generate heat) the liquid nitrogen and helium will quickly expand (~700 x) and fill the room. This is an asphyxiation hazard and cause "cold burns" very rapidly. Should a magnet quench while you are in the Magnetic Resonance Laboratory, leave the room quickly.
- 4) Do not attempt non-routine operations such as variable temperature, probe changes, or non-routine experiments without the proper training or without first contacting the NMR staff.
- 5) User are responsible for knowing the hazard ratings of all of the materials they are working with.
- 6) Avoid making contact with the magnet. Never lean against or pull yourself up using the magnet.

## VI. VnmrJ 4.2 Interface
















































- 1) Main Menu
- 2) User tool bar
- 3) Graphics control
- 4) Message area
- 5) Command line
- 6) Experiment selector
- 7) Graphics display
- 8) Parameter Panel
- 9) Spectrometer status
- 10) Temp, Spin, and lock info

### Mouse Usage

The left button of the mouse is the primary button. It is used to activate areas such as the command line. It also sets the left cursor (red line) and decreases the values when using the interactive shim and lock controls. The right button is used mostly to set the right cursor and increasing the values when using the interactive shim and lock controls. The middle button (or scroll wheel) is used to set the multiplier (1x, 10x, 100x) for the interactive lock and shim controls.

## VII. VnmrJ Button

-  Create new workspace
-  Open spectrum
-  Save spectrum (svf)
-  Display options
-  Cancel command
-  Abort acquisition (aa)
-  Layout 1
-  Layout 2
-  Light graphics scheme
-  Dark graphics scheme
-  Display FID
-  Display 1D spectrum
-  Use one cursor
-  Use two cursors
-  Show full spectrum (f full)
-  Reset to full display (ff)
-  Zoom in (zoom into the region between the two cursors)
-  Zoom out (zoom out to previous zoom level)
-  Zoom mode (use mouse to click and drag region to be zoomed in)
-  Pan and stretch mode (left click to pan spectrum, right click to stretch spectrum)
-  Show integral regions
-  Show full integrals
-  Hide integrals
-  Delete all integral regions (cz)
-  Cut integral regions (left click to set regions, right click to undo)
-  Integral level/tilt
-  Show/hide axis
-  Show/hide peak picking threshold


-  Show/hide peak frequencies
-  Show/hide integral
-  Enter phase mode
-  Redraw
-  return
-  Show/hide annotations
-  Select annotation
-  Peak annotation
-  Add text
-  Add line
-  Add arrow
-  Add square
-  Add oval
-  Add polygon
-  Add polyline
-  Add x-bar
-  Add y-bar

## VIII. Sample Preparation

Please use tubes that are at least 7 inches long. **DO NOT** use cracked, chipped, or broken tubes as this could damage the NMR probe. Use only plastic NMR tube caps on the top of the NMR tubes.

Solvent volume should be about 0.6 mL (about three fingers high in a 5 mm NMR tube). It is possible to use less volume (not advised), but the sample will need to be centered with the coil location shown in the depth gage and shimming may be difficult. Heterogeneous samples or samples with insoluble material will result in lower resolution spectra. Please avoid these types of samples.

## IX. Preparing for Acquisition

Log into your account on the spectrometer. Enter **e** on the command line or use the  button to eject the D<sub>2</sub>O sample in the magnet. Carefully remove the sample from the spinner and insert your NMR tube in the spinner by pushing it gently into the hole using a back and forth rotation until the tube reaches the bottom of the depth gauge as shown below. Do not force the tube into the spinner if it feels tight. Most breakages occur while putting the tube into the spinner. Always hold the spinner and sample by the top rim. Do not carry it by the NMR tube nor the lower part (the shaft).



Insert the tube in the spinner by pushing it gently along the hole while rotating it back and forth.

Images by Eugenio Alvarado, Department of Chemistry, University of Michigan



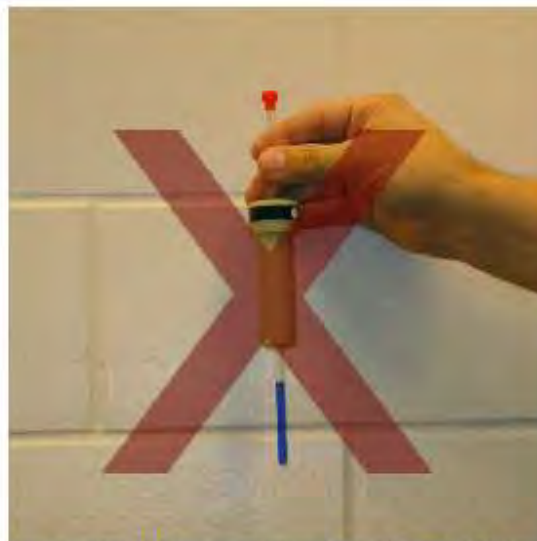
Hold the spinner from the top rim.



If you break your tube, you are responsible to thoroughly clean up the broken glass and any spilled chemicals. Failure to do so can result in suspension of NMR privileges.



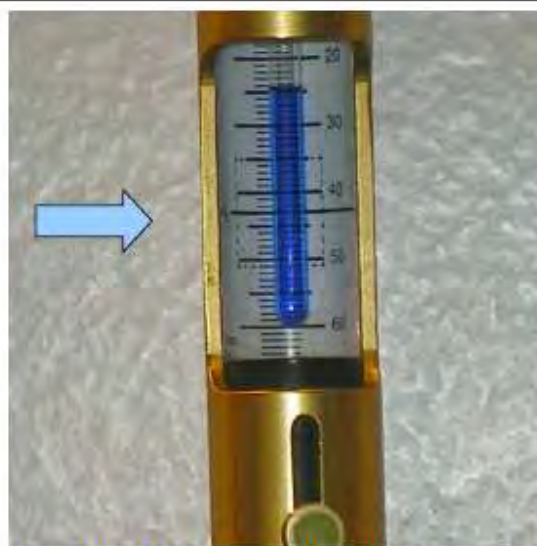
Do not hold or touch the spinner on the shaft



Do not hold the spinner from the NMR tube.

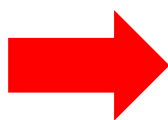


Do not insert the tube by forcing it in the spinner's hole, or when the spinner is mounted in the depth gauge.



If less than the recommended volume is used, center the solution's column with the center black line on the gauge's meter.

Images by Eugenio Alvarado, Department of Chemistry, University of Michigan



All samples should be prepared or modified (titrations for example) in your lab. Do not do anything sample preparation or manipulation in the Magnetic Resonance Laboratory.

Place your sample on the cushion of air. Don't release the sample until you are sure it is floating. Enter `i` on the command line or use the [Insert](#) button to lower your sample into the magnet. When the sample is in place you should hear an audible "clunk". With the sample in



the magnet it is time to lock and shim the sample. All UTSA NMR spectrometers are setup to perform automatic locking and shimming of samples.



On the Mercury Plus 300 make sure that the sample is spinning.  
On the DD2 500 the sample should **NEVER** be spinning.

- (1) On the sample info panel of the Start tab enter the sample name and hit enter. This will create the sample directory where all the spectra for the sample will be saved. It is advised that you name the directory after the notebook page the compound can be found on, ex. DJW-II-145. This will make it easier for you or more importantly, someone else to find the spectrum later. When starting a new sample click **Clear** before entering in the new sample name.
- (2) Select the solvent for the sample. The three most common solvents are on the panel. To select other solvents, use the pull down menu.
- (3) Click **Auto lock** to lock.
- (4) Click **Gradient shim** to auto shim the sample. For most applications this will provide satisfactory results. If this does not provide satisfactory result or you need to shim the higher order/non-spinning shims, see section **X. Shimming**.

The screenshot shows the 'Sample Info' panel of the NMR software. The 'Start' tab is active. The 'Sample information' section contains the following fields and controls:

- Operator: vnmr1
- Sample name: sample\_name\_here (1)
- Sample directory: (sample\_name\_here\_20160425\_01)
- Solvent: D20 (2)
- Buttons: DMSO, CDCl3, D20, Other
- Sample volume: 600  $\mu$ L
- Mass: 5.0 mg
- Notebook: IV
- Page: 125
- Clear button
- Email: you@email.com
- Comments: Add any comments you would like to this space.

Numbered callouts in red circles are placed over the 'Clear' button (1), the solvent selection (2), the 'Auto lock' button (3), and the 'Gradient shim' button (4).

## X. Shimming

The following is a good procedure for manually shimming an NMR. When optimizing a shim adjust the shim until the lock level is at a maximum. Make sure to try both increasing and decreasing the shim voltage.

The lower order shims ( $Z^1$ ,  $Z^2$ , X, and Y) have the biggest impact on line shape. Try to adjust these first (Steps 1, 2, and 5) before proceeding onto the higher order shims.

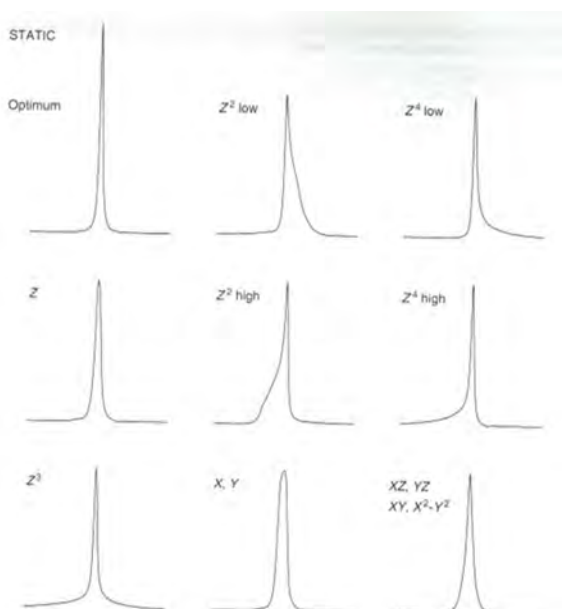


Some solvent (e.g. acetone- $d_6$  and  $CD_3OD$ ) take longer to respond to changes in the shims. You need to be patient when shimming with these solvents

- 1) Optimize  $Z^1$
- 2) Optimize  $Z^2$ . Repeat steps 1 and 2 until the lock is at a maximum.
- 3) Optimize  $Z^3$ . Repeat steps 2 and 1 until maximum lock is achieved.
- 4) Optimize  $Z^4$ . Repeat steps 3, 2, and 1.
- 5) Optimize X and Y in turn (just like with  $Z^1$  and  $Z^2$ ).
- 6) Optimize X and XZ interactively. Adjust  $Z^1$  to give max lock level.
- 7) Optimize Y and YZ interactively. Adjust  $Z^1$  to give max lock level.
- 8) Optimize XY interactively with X and Y.
- 9) Optimize  $X^2$ - $Y^2$  interactively with XY, X, and Y.
- 10) Repeat steps 1 and 2.

When the even ordered shims ( $Z^2$ ,  $Z^4$ ,  $Z^6$ ) are off, they will cause unsymmetrical distortions to the line shape. Odd ordered shims ( $Z^1$ ,  $Z^3$ ,  $Z^5$ ) will cause symmetrical distortions. The general rule is that the higher the order of shim, the lower down on peak the distortion is seen.


The following figure shows the peak shape when the different shims are incorrectly set. Recognizing these effects can make shimming easier.

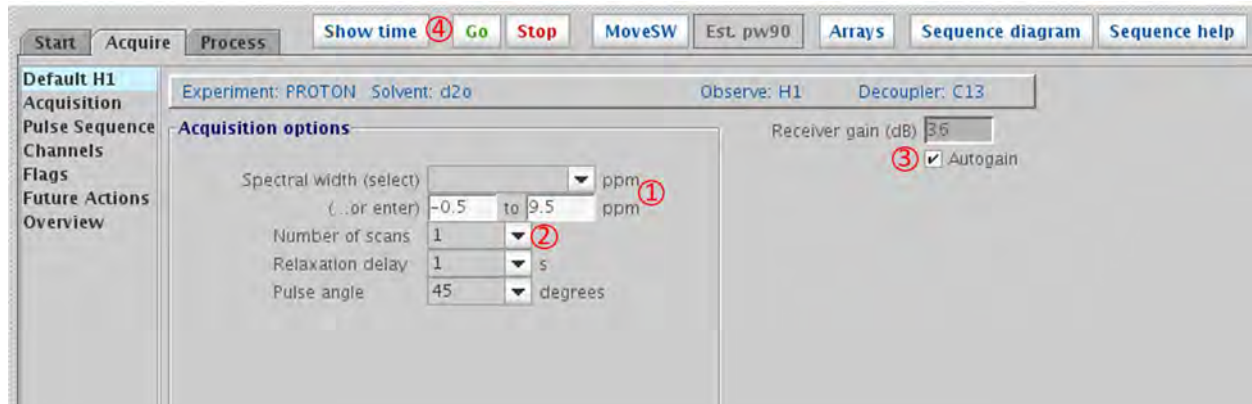




## XI. Acquiring 1D $^1\text{H}$ or $^{13}\text{C}$ spectrum

Select either PROTON or CARBON from the experiment selector.

- (1) On the acquire tab set the spectra width. For most applications the default is fine.
- (2) Set the number of scans.
- (3) Make sure the Autogain checkbox is checked.
- (4) Click on  to start the acquisition.




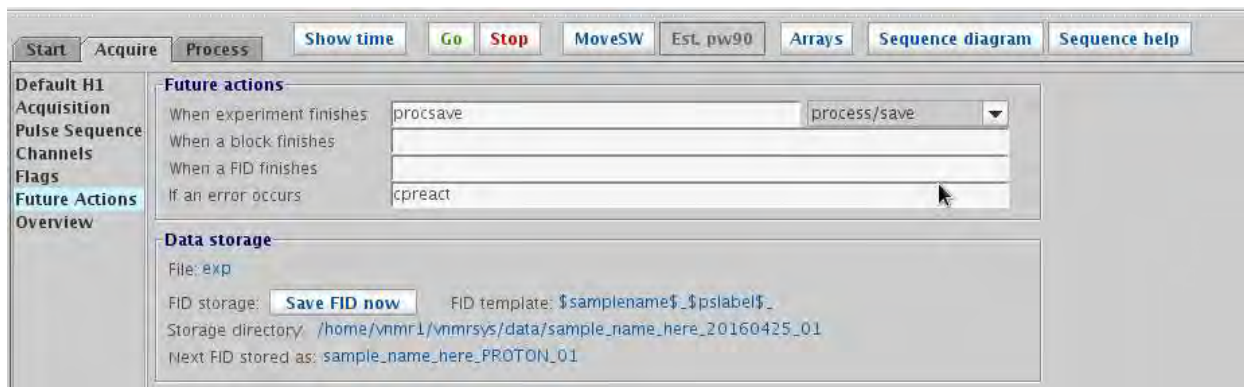
If the S/N of the spectrum is bad you can increase the number of scans and reacquire. Keep in mind that the S/N increase as a square root relationship. To double the S/N you have to take 4 times as many scans. To triple the S/N you have to take 9 times as many scans.





To see carbonyl and quaternary carbons it is better to use a Pulse angle of 30 degrees and a relaxation delay of 3-5 seconds. This will work better than taking more scans.

## XII. Saving the Data

There are many options for saving data. The easiest is to have VnmrJ automatically save it for you when the acquisition is complete. Under the Future Actions section of the Acquire tab make sure that process/save is selected from the drop menu of “When experiment finishes”. This will only happen if you use the  button. The spectrum will be saved in your “vnmrsys/data” folder in the directory that was set in step (1) of Preparing for Acquisition. **You must make sure that process/save is selected for before you run each experiment.**



If you forget to select process/save or don't want to autosave your data, the data can be saved by clicking on  in the Future Options section of the Acquire tab. The spectrum will be saved in your “vnmrsys/data” folder in the directory that was set in step (1) of Preparing for Acquisition.

You can also click on the save icon  and enter the name.

The final way to save the data is to type **svf** on the command line. You will then be prompted to enter the name that file will be saved as.

## Summary

1. Insert Sample
2. Enter sample information and select solvent
3. Lock the magnet
4. Shim the magnet
5. Select an experiment
6. Adjust spectral parameters
7. Start the acquisition
8. Save the data
9. Repeat steps 5-8 for all experiment for the current sample (e.g.  $^1\text{H}$ ,  $^{13}\text{C}$ , COSY, HSQC)

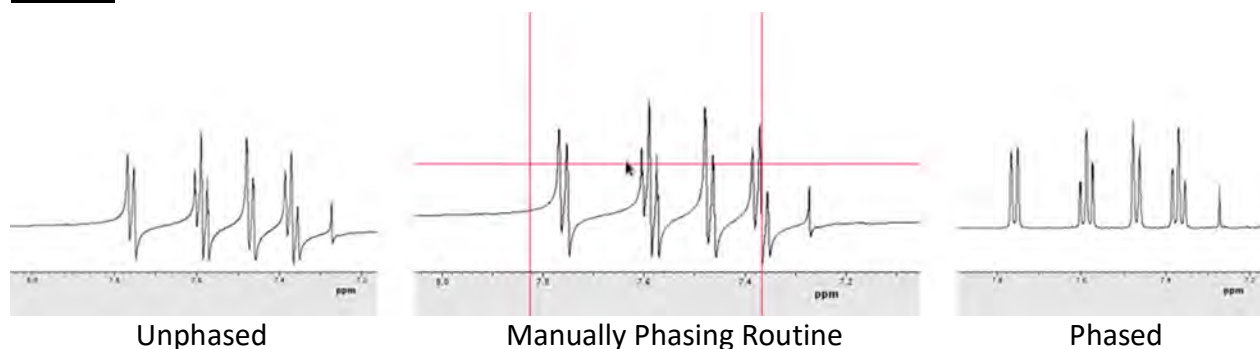
## XIII. Processing the Data

### Apodization


To improve the S/N of a spectrum apodization in the form of an exponential line broadening (lb) is often applied. For  $^1\text{H}$  spectra a lb of 0.3 Hz is used. For  $^{13}\text{C}$  a lb of 2 Hz is typical.

To apply line broadening type lb=# (# is the amount of line broadening you would like) on the command line and then enter **wft** to apply the line broadening.






### Phasing




To phase your spectrum type **aph** on the command line to do an automatic phase correction. The spectrum should go from looking the left spectrum, with the peaks being both above and below the baseline, to the right spectrum with all peaks being above the base line.

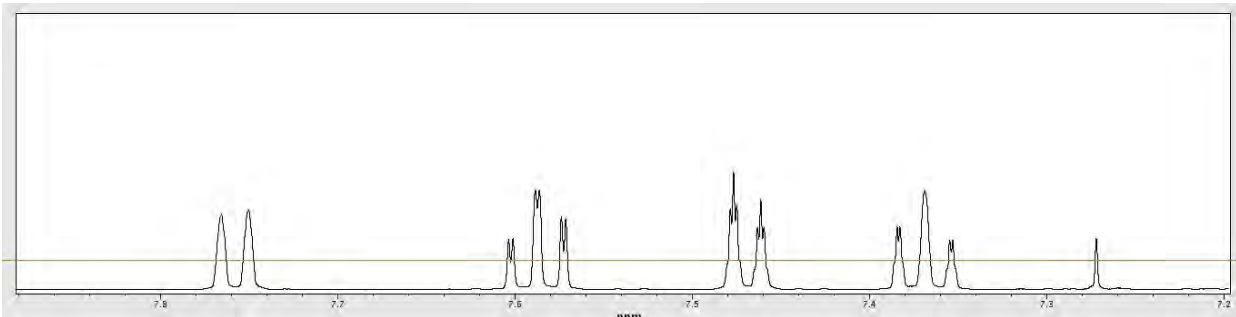
If this doesn't work, you will have to do a manual phase correction. Click on the Enter Phase Mode  icon. Left click near the left most peak. Left click and hold and drag the mouse up and down to phase the left side of the spectrum. Do the same on the right side of the spectrum until the phase is correct. Right clicking can be used in place of left clicking to get a fine adjustment of the phase.

### Integration

- 1) Click on the Show Integral Regions  icon. Enter **cz** in the command line to reset all integrals. Enter **dc** in the command line to set the baseline.
- 2) Click on the Define Integral Regions  icon. Left click on the left side of the signal you want to integrate. Left click again on the right side of the signal to set the integral region. Do this for all signals of interest. Right click to undo if you make a mistake.
- 3) Click on the Display Cursor(s) icon  to bring up the red cursor. Place the cursor over the peak you would like to use to normalize your integral values. On the integration menu of the Process tab enter the number of protons for the peak under the cursor in integral area. Click  to set the value. Click  to display the integral values.
- 4) To exit the integration routine, click on  followed by .

### **Peak Picking**

To label the peak chemical shifts click on the Show/hide Threshold  button to show the peak picking threshold line. Use the left mouse button to adjust the line so that the line is above all peaks of interest. Click the Show/hide Threshold button again to high the threshold line.



Click on the Show/hide peak frequencies  button to display or hide the peak frequencies.