

---

## SAMPLE PREPARATION

The quality of your spectrum is GREATLY affected by the sample preparation.

- Always use a clean and dry sample tube. A tiny scratch can destroy the high quality properties of the tube making it useless for high resolution experiments. For proper way of cleaning and conserving tubes visit Wilmad website ([www.wilmad.com](http://www.wilmad.com) - service/support nmr tube)
- The recommended sample volume for a 5mm tube is 700 $\mu$ L or 0.7mL of deuterated solvent. If you use a sample with a different volume it will be much harder to achieve a good shimming within a reasonable time as all the standards used to calibrate the machine had this volume. For a 5mm Shigemi tube the recommended volume is 150 $\mu$ L.
- By definition, when dissolved the sample should be free of any particulate material. Also avoid the inclusion of gas bubbles. Since dispersed or settled debris and bubbles will makes shimming very difficult take an extra time filtering and eliminating bubbles from your sample **before** trying to use it.
- Your sample needs to be free from impurities that can be easily removed, i.e., non deuterated water, acetone, methanol or acetonitrile. If when you record a  $^1\text{H}$  NMR spectrum these signals are present and larger than the residual solvent signal of the deuterated solvent been used you **MUST** remove these impurities since this has a **huge** effect on the sensitivity of your experiment (it affects the gain and hence the dynamic range). You can approach this problem from two sides. If you only need to get a proton (like with a flash column fraction), you can do a presat experiment. It is simple to set up and it takes only a minute to run. But, if you are going to collect any data that requires a long block of time, you need to go and get ride of the solvent. The easiest way to do this is to leave the sample in the NMR tube and remove the solvent under a stream of  $\text{N}_2$ . Redissolve the sample in a small amount of deuterated solvent and evaporate that off again. Add more deuterated solvent and check the proton spectrum, if the solvent impurities are still present repeat.

Always remember because these experiments involve a fourier transform that to double your signal-to-noise ratio requires four times as many scans. So if you can't see anything after 1h you are not going to have much to look at after 4h, likewise if you can not see anything after 2h it is not going to improve a lot after 8h. So on long experiments check your data after an hour or so and if can not see any correlations you might as well stop the experiment.

---

## LOGGING IN:

The computer will first ask for your LOGIN (enter your user name in lower case letters), and then your PASSWORD. A login and password will be given to you upon completion of the training.

After login, you will enter the command “**vnmr**” in the terminal window. Once everything has started the screen will appear as shown in Figure 1

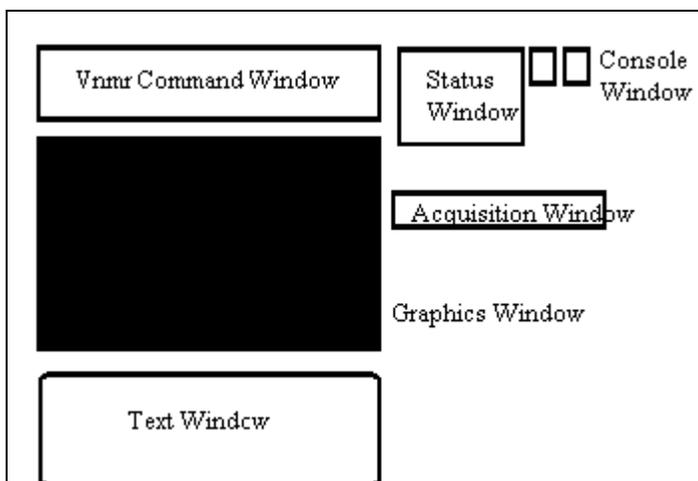


Figure 1 – Desktop after vnmr is initialized

**Command Window:** This window is used to enter commands for Vnmr. This can be done by typing commands or by using one of the many buttons presents. On the upper part of this window is where status and error lines are showed.

**Graphics Window:** Spectra and pulse sequences are displayed in this window.

**Text Window:** Also called **dg** windows, this is the place where parameters and messages are displayed. In its right side is a toolbar where different sets of parameters can be selected (acquisition, decoupler, sequence, etc.) The text option is the one most Vnmr users are familiar with it.

**Status Window:** Displays the status of the spectrometer, e.g. which experiment is in progress, number of scans, time remaining, sample temperature and number of experiments queued for acquisition.

**Acquisition Window:** This is a pop-up window which allows the user to modify the shims and deuterium lock parameters. This window is not available when acquisition is in progress.

**Terminal (Console) Window:** This normally appears as a small icon and is opened by clicking on it. Generally it plays little role in the routine use of Vnmr. However, it is very important as system messages are displayed in this window if an error occurs. If you encounter difficulties in acquiring data you should check this window before contacting the administrators for assistance.

---

## LOCKING

The lock system maintains the frequency stability of the spectrometer as the static field generated by the superconducting magnet drifts slowly with time or changes due to external interference. It is essentially a deuterium NMR spectrometer that monitors the frequency of the solvent deuterium resonance and adjusts the magnetic field to keep the frequency constant. The exact frequency of the deuterium signal is different for each solvent but the spectrometer uses a list of the deuterium frequencies stored in one of the configuration files to adjust its parameters to accommodate these differences.

Besides adjusting the lock signal, three other parameters need to be adjusted: lock power (radio frequency power), lock gain and lock phase. If too much power is applied to the deuterium nuclei, they will saturate as those nuclei will not be able to dissipate the energy and this will result in line broadening and variation of the signal amplitude. Saturation level will vary between solvents, e.g. as acetone has 6 atoms of deuterium per molecule its power level will be much lower than for chloroform that has only 1 atom. A too high lock gain will lead to over amplification of the lock signal, thereby causing excessive noise. The **lock phase** must be correctly set for efficient operation of the lock. The lock system feedback mechanism actually monitors the dispersion mode signal of the deuterium, thus correct phase adjustment is required to make this so. The lock phase does not vary from sample to sample, but does vary slightly with lock power. If the phase is grossly miss-set, the instrument will not lock at all. Indeed, if the phase is out by 180 degrees, a situation of "anti-lock" is produced.

### Steps:

1. Check the setting of the "solvent" parameter.

Select [**Setup**]. [**Nucleus,solvent**]. Choose [**H1**] or [**C13**]

2. Check that the temperature is set correctly in this experiment.

Type "**temp**" to see to what temperature the machine is set up. If necessary change for the new value.

3. Enter the command "**su**". Whenever you set up a new experiment or make changes to an existing set of parameters your changes are not immediately transferred to the acquisition computer but stored in a temporary file. When you have finished setting up your new parameters the changes must be transferred to the acquisition hardware by the command "**su**". Only after typing this command will be initialized your new parameters.

4. Open the Acquisition window by selecting [**Acqi**]. See Figure 2

This window allows insertion and ejection of sample. It also allows adjustment of lock, shims and spin parameters. These adjustments are done by sliding the bar correspondent to the parameter being changed or by clicking on the buttons like [-16+]. Using the second method is done with your mouse buttons. Clicking the button with left mouse button will decrease the parameter by the value on the button. In a similar way, clicking with the right button will increase the parameter by the value on the button.

5. Click on the [**Lock**] Button.

6. Select Sample: [**eject**].

Remove solvent from the spinner and insert sample to be analyzed. Use the sample depth gauge to position it correctly. Remember that glass do not bend well... So, be careful to position your tube strait when inserting it in the spinner. For normal

sample, the bottom of the tube should be in the bottom of the depth gauge and, for shorter sample, it should be in the center of the traced square of the depth gauge.

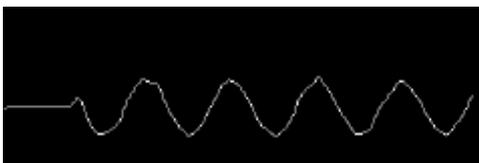
7. Select [**insert**] to lower the sample into the magnet

8. Select Lock: [**off**] and Spin: [**on**]

Adjust value of spin for 20.

9. Increase the "Lock Power" and "Lock Gain" until you can see the lock signal.

It should appear as a sine wave. If a sine wave is not visible increase lock power and lock gain until you can see something like in the figure bellow. The number of peaks seen in the screen is directly related to how far the value is from the correct value for locking.



10. Adjust the value of Z0 to get maximum intensity. The final signal should have no modulation and only a small decay.

Adjust **Z0** in steps of 1 or 4 (300MHz) or 16 or 64 (500MHz) aiming to a increase in the wavelength of the wave (less peaks visible). Keep adjusting until one square wave is seen, turn Lock [**on**].



11. Click the lock "ON" button.

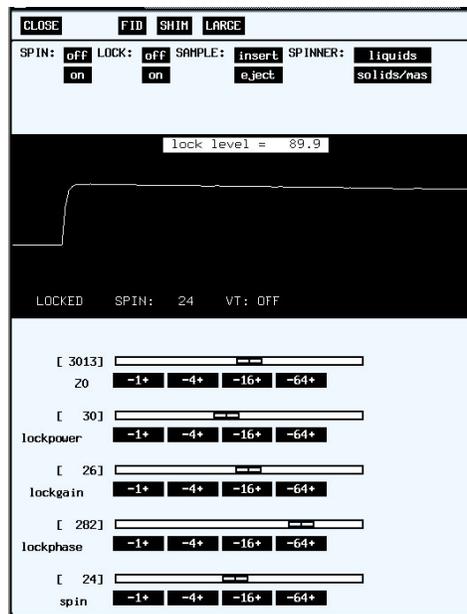


Figure 2 – Acqi window

Reduce **Lock Power** and **Lock Gain** until the lock level is <100%. Ideally **Lock Power** should be approximately half of **Lock Gain**.

12. Adjust the "Lock Phase" to maximize the lock level.

---

## SHIM

To create a homogeneous (no variation) magnetic field over the sample, coils of wire (**SHIM COILS**) are placed in the vicinity of the sample, and currents (**SHIM CURRENTS**) are passed through them to create various gradients of any desired strength. The idea is to adjust the shim currents so as to cancel any gradients in the NMR sample as accurately as possible. This procedure is called **SHIMMING THE MAGNET**.

The frequency of an NMR line is directly proportional to the strength of the magnetic field. If the strength of the magnetic field varies by some percentage over an NMR sample, the resonance frequency will vary by the same percentage, which will smear out the NMR lines and possibly distort their shape. If you want to see a proton line as sharp as 0.3 Hz on a 300 MHz spectrometer, the field must vary over the sample by less than (0.3 Hz/300,000,000 Hz) or one part in  $10^9$ . Since the diamagnetic susceptibility of organic solvents is of the order of  $10^{-7}$ , gradients in the NMR probe will change of the order of 100 parts in  $10^9$  when you remove or insert samples. Replacing one sample with another containing a different solvent (or filled to a different height - or inserted into the spinner by a different amount - or in a slightly different NMR tube, or changing the sample temperature, etc.) can cause a noticeable change in the NMR line shape and width. Paramagnetic samples obviously cause much larger changes.

In order to obtain satisfactory spectra, any operator of NMR spectrometer must be able to shim out the relatively small gradients caused by change of sample and by sample temperature. This must be done fairly accurately, or lousy spectra will be produced. This routine shimming can be simple, fairly fast, and consistently accurate if done properly. It can be time consuming and produce inconsistent results if done wrong. **So, proper shimming strategy is important, because it helps you to get a good shim fast.**

Shimming is simple in principle, just adjust the shim currents until you have cancelled out the gradients over the NMR sample as much as possible. In practice, it is a bit more complicated and tedious. Part of the problem is that the effects of many shim controls interact, so a bad setting of one shim control will prevent you from finding the exactly correct setting of another. A related problem is false optima: You can find some combinations of shim settings which are not good, but changing any one shim by a small amount actually makes things worse. The most commonly-used criterion for shimming - the height of the lock-signal level - is rather insensitive to some shims, almost never points the way to the best possible shim, and is just plain wrong under some conditions. It helps to be able to identify which shims need to be adjusted, how much and in what direction and in what order to adjust them, and to know when to quit.

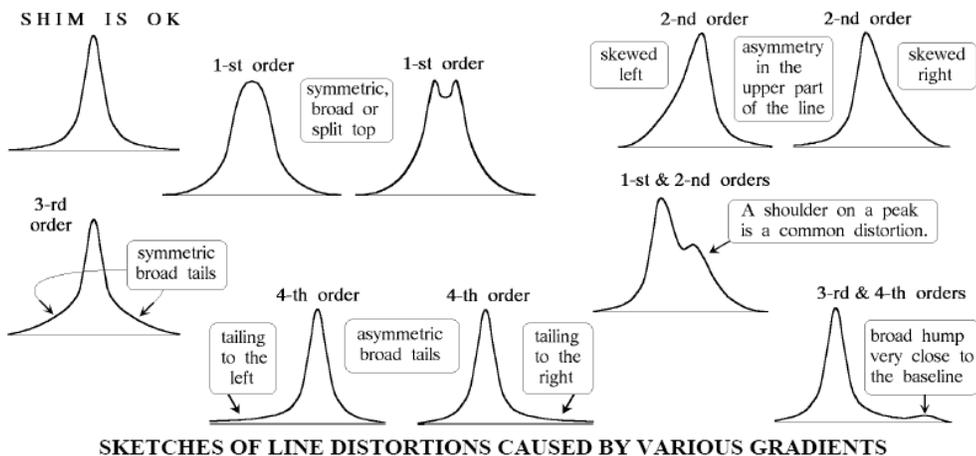
The shim controls on spectrometers can be divided into different classes, depending on the type of distortion which each gradient produces in the NMR lines, and depending on whether or not spinning the sample reduces the distortion. From the shape of the NMR line(s), you can get a good idea of which gradients are bad. In general, odd-order gradients cause symmetrical distortions of NMR lines, and even-order gradients

produce skew distortions. Low-order gradients distort the entire NMR line, while high-order gradients distort mainly the bottoms ("feet" or "tails") of the lines. The higher the order of the gradient, the lower down on the line you have to look to see the distortion.

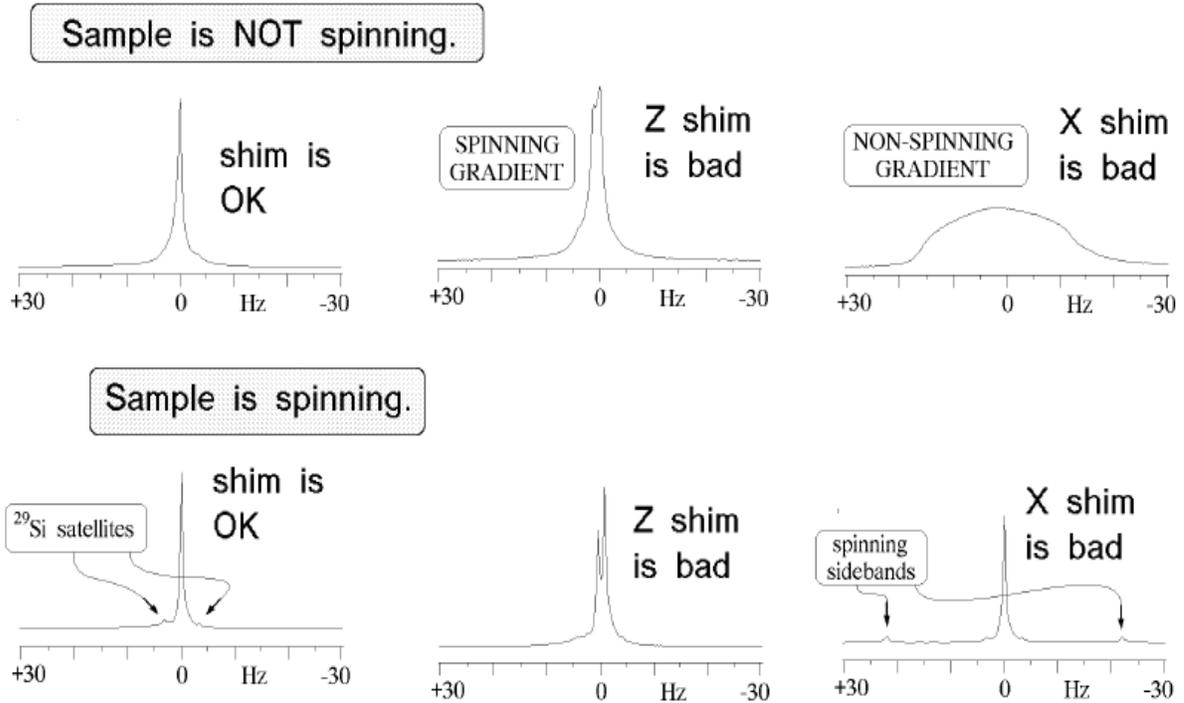
In the case of even-order gradients, the shape of the NMR line will tell you which direction you want to move the shim-control to improve things. The skew (or "tailing") moves from one side of the NMR line to the other as an even-order shim is moved from a bad setting, through the correct setting, and further on to a bad setting on the other side. On the spectrometers, the correct direction to move the shim control is toward the opposite direction of the "tailing" in the line for both the  $Z^2$  and  $Z^4$  shims.

Shim name	Gradient order	Interaction order
Z0	0	-
Z1	1	-
Z2	2	$Z^1$
Z3	3	$Z^1 (Z^2)$
Z4	4	$Z^2, Z^0, (Z^1, Z^3)$
Z5	5	$Z^3, Z^1, (Z^2, Z^4)$
Z6	6	$Z^4, Z^2, Z^0 (Z^1, Z^3, Z^5)$
X1	1	Y, ( $Z^1$ )
Y1	1	X, ( $Z^1$ )
XZ	2	X, ( $Z^1$ )
YZ	2	Y, ( $Z^1$ )
XY	2	X, Y
$X^2-Y^2$	2	XY, (X, Y)
X3	3	X
Y3	3	Y
$XZ^2$	2	XZ, (X, Z)
$YZ^2$	2	YZ, (Y, Z)
ZXY	3	XY, (X, Y, Z)
$Z(X^2-Y^2)$	3	$X^2-Y^2, (X, Y, Z)$
$Z^3X$	4	
$Z^3Y$	4	
$X^2Y^2$		
$Z^2XY$		

The following figure give an idea about distortions on different shims and a good shimmed peak.



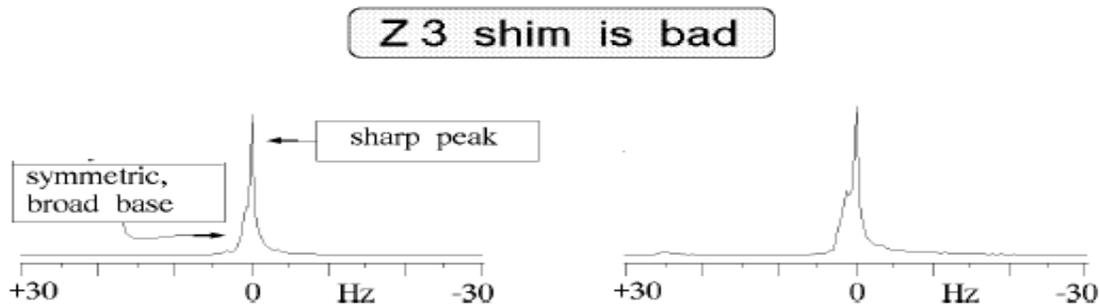
**Effect of sample spinning:** Distortions of the line shape which are caused by spinning gradients are not changed, but distortions caused by non-spinning gradients are greatly reduced.



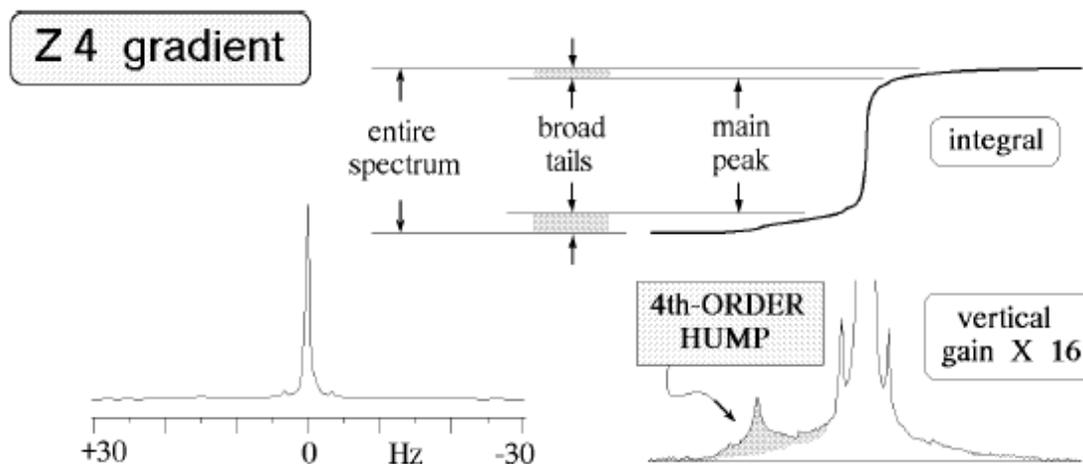
**Second-order gradients** produce **skew** distortions visible in the **upper part** of the line. The shape of the line is the most sensitive and unambiguous indicator of an imperfectly-adjusted second order shim and it tells you which direction to change the shim. The lock level is not good at detecting a slight error, or at distinguishing between a second-order shim error from a third-order shim error. The splitting of the line is caused by interaction with a first-order gradient - Z, in this case.

Error! Objects cannot be created from editing field codes.

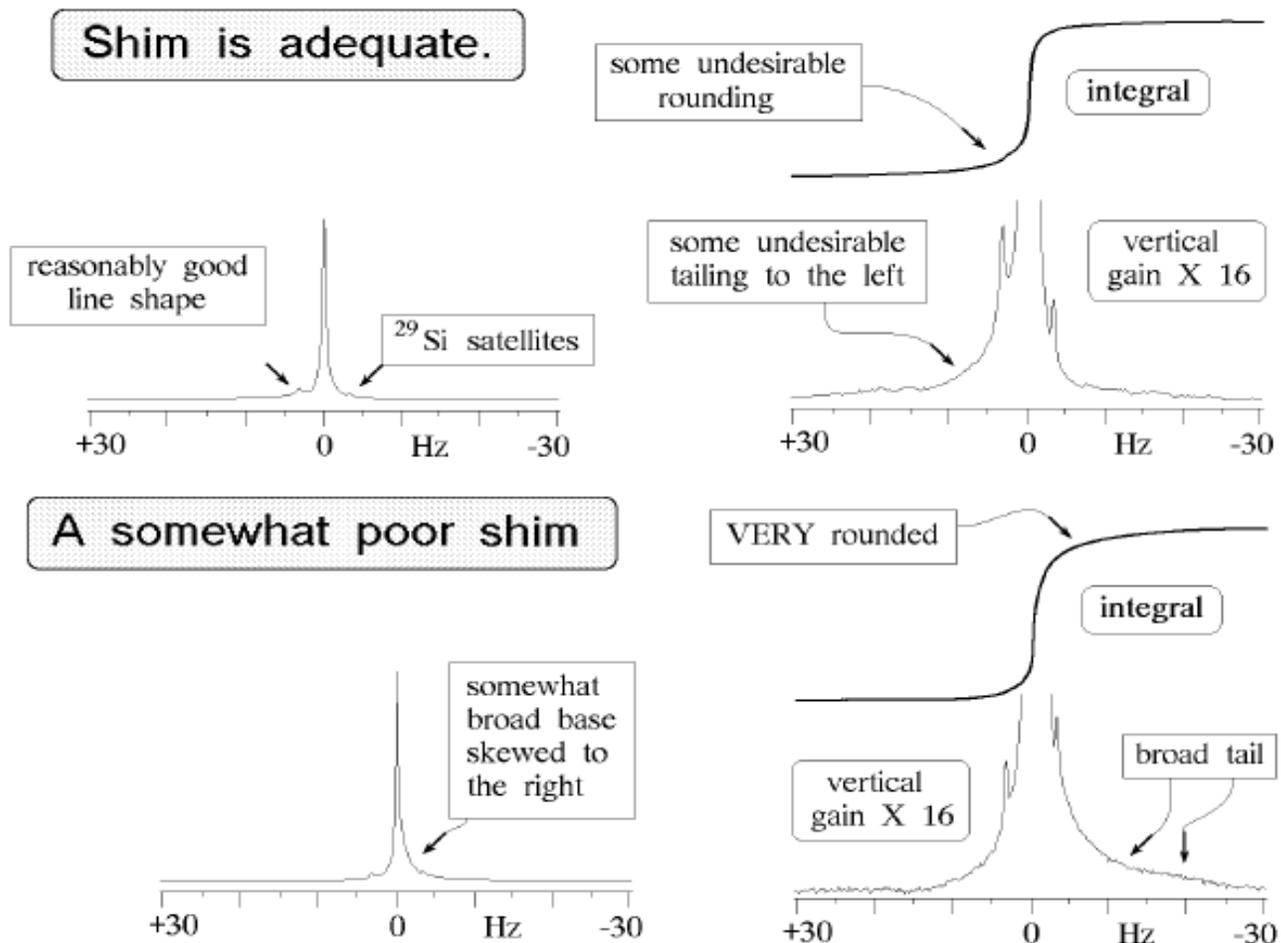
**Third-order gradients** produce **symmetric** broadening at the **bottom** of the NMR line.



**Fourth-order** gradients produce **skew** distortions of the "tails" or "feet" of the NMR line. If the first- and second-order shims are optimized, the broad 4-th order hump can be so close to the baseline that it is difficult to see it unless one observes the integral or uses high vertical gain. This particular line has a fourth-order hump on the left, which has been shaded for clarity. It also appears to have some symmetrical fifth-order broadening (very close to the baseline).



**In most cases, it is far more important to have a good line shape than to have the upper part of the line be as narrow as possible.** The advantages of good line shape are better sensitivity (in most cases), the ability to accurately compare integrals of lines which are closer together, and the ability to apply resolution-enhancement techniques. It's rather easy to get extremely good homogeneity over perhaps 70% of the volume of the sample, and lousy homogeneity over the rest of the sample. The consequences of this mistake are shown below. Don't be deceived by the impressive sharpness of the tops of the NMR lines. **Sensitivity is lost** by the "leakage" of part of the integral of the signal into the broad base and tails of the line, and it is **impossible to accurately compare integrals** of closely-spaced lines, because the tails overlap. (Looking at it another way, notice that the integral of the line doesn't level off until it's about 25 Hz to the right of the center of the line). The **Gaussian resolution enhancement** technique presupposes that the line shape is pure Lorentzian, and it **does not work well** with badly distorted line shapes such as this one.



### Who needs to shim which gradients? When?

The first- and second-order spinning shims need to be adjusted for each sample. For routine use of most nmr spectrometers, this is all that is necessary. **Operators should become very familiar and comfortable with this procedure before attempting to adjust any additional gradients.**

In addition to the first - and second-order spinning shims, some higher order spinning gradients frequently need to be adjusted in those situations where the best possible proton resolution is required – when it's very important to make the broad feet around proton lines narrower. These adjustments **really** do have to be made on each individual sample. On the 300 MHz spectrometer, you'll need to adjust the first 3 spinning shims ( $Z$ ,  $Z^2$ , and  $Z^3$ ). On the 500, you'll need to adjust the first 4 spinning shims – ( $Z$ ,  $Z^2$ ,  $Z^3$  and  $Z^4$ ). For that you have two choices: you can do that manually or you can use the automatic gradient shimming in CustomQ.

It is sometimes desirable to run a sample with the spinner off. 2D and selective 1D experiments give better results when run non-spinning. In this case, you need to adjust **all** of the first-order gradients ( $Z^1$ ,  $Z^2$ , X, Y). All 4 of these gradients must be adjusted with the spinner off, as well as the second order spinning gradient.

The rule of thumb for non spinning gradients is that if you turn off the spin and the signal decrease more than 10% you need to touch up them.

Spinning sidebands in spectra probably indicate that the first-order non-spinning shims require adjustment. This can be done by any experienced operator who is familiar with the procedure. **Normal users of the spectrometers will not need to shim any other gradients.** The sample-to-sample changes in the high-order gradients are negligible, and they drift only very slowly with time.

## Common Problems with Shimming

Neglecting to touch up the lock phase before making final shim adjustments produces unreliable results from a lock-level shim. It is simply not possible to set the lock phase consistently with sufficient accuracy while looking at the sweep pattern; furthermore, the lock phase will change whenever the lock transmitter power is changed, which happens automatically in many spectrometers when the lock is engaged. Inconsistencies in the phase settings produce inconsistencies in lock-level shimming results.

Sometimes the NMR lines are symmetrical and % Hz wide, sometimes they tail off to the right or left, and sometimes the lines are 1 Hz wide or broader. Even if you do your final shimming on the proton FID and line shape, a very misleading lock-level response will make the shimming process unnecessarily tedious and time-consuming.

The ultimate criterion for a good shim is the observed nucleus, not the deuterium lock signal. The highest possible deuterium lock-signal level is usually *not* obtained at the same shim settings as the best shape and narrowest width of a  $^1\text{H}$  or  $^{13}\text{C}$  line in the same sample. The difference is small for many samples, moderate for some samples, and spectacularly huge for a few unusual samples. For adjusting high-order gradients, the lock-signal-level criterion is particularly likely to give misleading indications, which can lead to very artistic results.

Changing a probe produces strong, complicated gradient changes, so it's necessary to "dial in" the appropriate "standard" shim settings for each probe before using it. Forgetting to do this is actually a very uncommon problem, but it's mentioned here because you can waste a huge amount of time and be very frustrated while trying to get a good shim if the high-order gradients are off. If you ever have serious trouble getting a good shim within a few minutes, then stop, re-set the shims to their "standard" values, and proceed from there.

Different magnets do respond differently, depending on such things as manufacturing details, the initial adjustments which were made when the spectrometer was installed, and on sample characteristics. People who have only run one particular type of sample on one particular instrument sometimes find that they can't get a good shim on some other instrument. Frequently, the problem is that the particular shimming strategy that they had been using is not generally optimum, even though it happens to work very well most of the time with a particular class of samples on one particular spectrometer.

## ROUTINE SHIMMING

**PRELIMINARY STUFF:** Put your sample in the spectrometer. Select solvent and nucleus to be observed. Set “nt = 1”. Lock and phase your sample. Adjust the lock level to about 80 if possible.

### **Spinning Shims**

1. Click on the *ACQI* button in the Main vnmr window.
2. Click on the *SHIM* button and set spin to on.
3. Maximize lock level with Z1
4. Maximize lock level with Z1 and Z2. Do this by making a change in Z2 followed by maximizing Z1 again. Continue in this manner until you can no longer increase the lock level.
5. Acquire the spectrum and analyze peaks for imperfections (use the shimming figures above as guide).
6. If the lines are symmetric and sharp your work is done! Congratulations!! If the lines are not symmetric or unusually broad at the base you need to adjust Z3, Z4 or the non spin shims.
7. If you need to adjust Z3, do it in the same manner that for Z1 and Z2 in step 4. Changes in Z3 may affect Z2 so after shimming Z3, repeat step 4.
8. If Z4 needs to be adjusted, look at which side of the peak the asymmetry appears. If the asymmetry is large (Z4 is far off), change Z4 by a considerable amount to try to push the asymmetry to the other side of the peak. This provides two important pieces of information: confirms that Z4 is the problem if the asymmetry moves. Indicates what the actual value for Z4 should be when Z4 is changed. Since you know the values that caused it to be on either side of the peak, the correct value must be between the two extremes.
9. Set Z4 to the value that produces no asymmetry.
10. Since Z4 affects all the shims bellow, repeat steps 1 to 7.

### **Non spinning shims**

1. Write down the lock level, set spin to off, and write down the lock level.
2. Maximize lock level with X
3. Maximize lock level with Y
4. Maximize lock level with X and Y. Do this by making changes in Y followed by maximizing with X again. Continue in this manner until you can no longer increase the lock level.
5. Maximize lock level with X and ZX.
6. Maximize lock level with Y and ZY.
7. Repeat step 3.
8. Maximize lock level with XY and ZXY
9. Repeat steps 4, 5 and 6.
10. Set Spin to on and acquire a spectrum.
11. If the lines are not symmetric or are broad repeat steps for spinning shims.



# Varian NMR Instructions - 1D Quick Reference

Login:

## Insert sample, autolocking, and shimming

- 1) To eject the standard sample type “e” (eject)
- 2) Center sample in depth gauge and insert into magnet
- 3) Type “i” (insert) to lower sample
- 4) In the Tcl-dg panel under “Setup experiment”
  - a) **select solvent**
  - b) select “**Find Zo**” (this autolocks)
- 5) Go back to “Setup Experiment”
  - a) Under Shimming select “**2H-grad**” (gradient autoshimming)

## Acquiring a Proton Spectrum : Setup and Acquisition

- 1) Select [Main Menu] - [Setup] - [Nucleus, solvent] - [H1] - [solvent].
- 2) Turn off autolocking (**alock='n'**) and autoshim (**wshim='n'**)
- 3) Set “**bs=2**” (bs= block scan or minimum number of scans before you can look at spectra)
- 4) Set “**nt=128**” (nt = total number of transients or number of fid collected)
- 5) When ready type “go” (starts experiment)
- 6) type “**wft aph**” after appropriate number of scans (this is set by bs value)  
(wft aph = weighted fourier transform and autophase spectra)  
If spectra has very bad signal-to-noise then use the “weighted function to improve”
  - a) type “**lb=0.8**” (line broadening – this will obscure small proton-proton couplings) and “**gf=0.8**” (Gaussian function – mathematical shift of the FT to amplify the portion of the FID with better S/N at the expense of your resolution)
- 7) Spectra will continue to acquire till “ct” (current transient) = “nt”
- 8) Repeat step 6 till adequate S/N then type “sa” (stop acquisition)

**Remember (for FT experiment  $S/N = (nt)^{1/2}$ ; so to double your S/N requires 4x as many scans and therefore 4x as much time. If you get to 256 scans and have poor S/N either increase the concentration of the sample or type a high-field NMR.**

- 9) To add a title “Text(‘A Title of your choosing’)”
- 10) To save data:
  - a) **Make sure you are in the correct directory! i.e**  
“/export/home/Your\_group\_name/vnmrsys/data/yourname”
  - b) If not then: [Main Menu] - [File] - [Data] and follow the buttons to change into your directory
  - c) [Save FID] or simply type **svf** [return] in your data directory.
  - d) Supply a filename when prompted. (The filename can't contain spaces)

**ds** will redisplay your spectrum.

## Loading Data:

- 1)[Main Menu] - [File] - [Data]
- 2) Highlight filename and select [Load FID].
- 3)Type **wft**.

## Data Processing and Analysis

Interactive Display Mode (ds command): To adjust height of spectra, position, or zoom

- Start this mode by typing **ds** or by selecting **[Main Menu]** - **[Display]** - **[Interactive]**.
- Left and right mouse buttons control the red vertical cursors.
- Middle mouse button controls vertical scale of spectrum (**vs**) or integral (**is**), if displayed.
- Type **vsadj** / **isadj** to automatically adjust vertical scale of spectrum / integral.
- **[Cursor]**/**[Box]** buttons toggle between 1 and 2 vertical cursors.
- **[Expand]**/**[Full]** buttons allow zooming in and out on regions of the spectrum.

### Phasing:

- Type **aph** for automatic phasing.
- For manual phasing in the interactive display mode, select **[Phase]**.
- Click on a peak near the right edge of the spectrum and drag up and down with the left (coarse) or right (fine) mouse button to phase this peak (adjust **rp**).
- Click on a peak near the left edge of the spectrum and drag up and down with the left or right mouse buttons to phase this peak (adjust **lp**).
- Select **[Box]**/**[Cursor]** to get out of phase mode.

### Referencing:

- Find reference peak (TMS or solvent) and click on it to move the red vertical cursor to that position.
- Type **nl** to move to the top of the nearest line.
- Type **rl(.\_p)** with the value for your solvent (i.e. if your solvent is CDCl<sub>3</sub>, type **rl(7.24p)**).

### Peak Picking:

- Select **[Th]** button to display yellow horizontal line.
- Use left mouse button to drag yellow line to set threshold for peak picking.
- Select **[Th]** again to get out of threshold adjustment mode.
- Type **dpf**, **dpf('pos','top')** or **dll** to list peaks.

### Integration:

- Select **[Int]** to display integral trace (green line).
- Select **[resets]** to start integral reset mode.
- Click with the left mouse button on both sides of each peak of interest to cut integral regions.
- If you make a mistake, click with the right mouse button to remove the nearest integral reset.
- To start over, type **cz** to clear all reset points.
- Select **[Box]**/**[Cursor]** to get out of integral reset mode.
- Type **dpir** or **dpirn** to display integrals below spectrum. (**vp** must be =12).
- (optional) Type **bc** for baseline correction. *All* peaks must be integrated for this to work properly.
- (optional) Select **[lvl/tlt]** button to adjust slope and bias of integral:  
Click on integral on left side of spectrum and drag up or down to level the integral.  
Click on integral on right side of spectrum and drag up or down to level the integral.  
Select **[Box]**/**[Cursor]** to get out of lvl/tlt mode  
Repeat if necessary.

Plotting: Type: "**pl pscale pltex ppa page**" add **ppf** before **page** to print peak positions

- Use **text** command to set title for plot: **text('title of plot')**.
- Automatic plotting is available by typing **plot**.

### **Commands for manual plotting**

**pl** - plot spectrum and, if currently displayed, the integral.

**plww** - plot a series of spectra taken in an arrayed experiment in whitewash mode.

**pl('all')** - also plots a series of spectra from arrayed experiments, not whitewashed.

**pscale** - plot axis under spectrum. To change the units on the axis type **axis='h'** for Hertz or **axis='p'** for ppm.

**pap** - plot all parameters in the upper left of the page.

**ppa** - plot a short list of parameters in the upper left corner of the page.

**ppf** - plot peak frequencies above spectrum with a line going down to the peak.

**ppf('top')** - same, but peak frequencies are all plotted along the top of the plot.

**ppf('pos', 'top')** - same, but plot only positive peaks .

**vp=vp+80 ppf('top')** **vp=vp-80** - same, but plot with short lines that don't go all the way down to the top of the peak.

**pir** - plot integrals under the spectrum. **vp** (vertical position of the spectrum) must be at least 12 for this to work.

**pirn** - plot normalized integrals under the spectrum. Again, **vp=12**

**page** - send plot to the plotter. The output from all plotting commands goes on the same plot until the **page** command is typed.

**page('clear')** - delete everything that has been plotted so far instead of printing it.

**pll** - plot a list of peak frequencies in ppm and in Hz. Do not do a **pap** or **ppa** or **page** if you do this command.

**pli** - plot a list of integrals. Do not do a **pap** or **ppa** or a **page** if you do this command.

**text('title of plot')** - set plot title.

**pltext** - plot the title set by the **text** command in the upper left of the plot. The text will also be plotted with the **pap** and **ppa** commands.

**Example: pl pscale pl text page**

#### Parameters:

**wc** - width of chart in mm. Determines the width of the spectrum on the page.

**sc** - start of chart in mm. Distance of right edge of spectrum from right edge of page.

**wp** - ppm range of the plot.

**sp** - start of the plot in ppm.

**vp** - vertical position of spectrum in mm. Moves spectrum up and down on the page.

**wc2** - height of chart in mm. Usually set equal to **wc2max**. Determines where top is in **ppf('top')** command.

**vs** - vertical scale of the spectrum.

#### **Printing**

-- Anything output to the VNMR text window can be printed: **printon commands printoff**, eg: **printon dg dg1 dg2 dgs printoff** will print all the parameters to a page.

#### **Exiting VNMR**

-- *Reinsert the solvent sample and lock and shim on it!!*

-- Type **exit** on VNMR command line or use permanent menu button: **[Exit VNMR]**.