

Shimming and locking

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After preparing the sample and loading it in the magnet, the acquisition of a routine 1D NMR spectrum involves a few steps:

- loading and setting parameters,
- establishing the lock,
- shimming and
- starting the acquisition.

After loading your sample in the magnet to take a spectrum, you have to adjust several dozen acquisition parameters like the spectral width (sw), acquisition time (at), transmitter offset (tof), relaxation delay (d1), pulse width (pw), transmitter power (tpwr), pulse sequence to be executed (seqfil), etc. All these parameters and their default values are contained in parameter files and are loaded in the spectrometer when you select a new experiment to measure from Vnmrj's menu. Luckily, for routine applications you only have to change a few.

In order to take a high resolution spectrum, the magnetic field has to be stable and homogeneous. If the magnetic field strength changes during the course of the acquisition, the signals will appear broadened and the spectrum will have poor resolution. To keep the field stable, the spectrometer has a mechanism that corrects the field's drift as it occurs ("the lock system"). The drift is measured using the absorption frequency of the solvent's deuterium resonance (the lock signal). As this frequency changes due to a change in the field, an electric current is applied to a coil in the magnet that increases or decreases the field's strength to keep the signal in the same position and the field constant. To continuously monitor the deuterium signal, the spectrometer has a dedicated deuterium spectrometer. When you "lock your sample", you tell the lock system to monitor the solvent's signal. But before you attempt to lock your sample, it is first necessary to set appropriate lock parameters: Z0, power, gain and phase. Unfortunately, these parameters are often not well understood by the user. The following are very brief descriptions of what these parameters are.

The lock field strength, Z0, controls the actual magnetic field strength at which the signal from the deuterated solvent resonates. It should remain constant for a particular solvent and magnet but the value changes slowly over long periods of time (months) as the magnet's field drifts. It should be adjusted so the lock frequency is in resonance with the solvent's absorption frequency. You do this by changing Z0 until the lock signal appears on screen as a flat, zero frequency sinusoidal signal. Usually, it is not necessary to be exactly on resonance, but you should be close. The *lock power* controls the radiation's power that is applied to the solvent's deuterium signal. Low powers give very weak signals and a poor, noisy lock; while too high powers produce signal saturation leading to lock instability. The optimum power is determined mainly by the relaxation time of the solvent and probe design. For example, in the Inova 400 the optimum power for CDCl₃ is about 24 and that for acetone-d₆ is about 6; using a power of 24 for acetone will certainly lead to severe saturation, instability and shimming will be impossible. The *lock gain* provides amplification to the lock signal. Its value is not really important as long as a lock intensity of at least 60% is maintained during acquisitions. The *lock phase* is mainly determined by the electronics and to a lesser extent by the solvent. Usually it is not critical to optimize it as long as an intense lock is produced. However, if you are unable to lock, make large changes (of ~20 units) and see if it helps. Optimize it if needed.

Instead of having to remember and enter appropriate parameters and to simplify the operation of the spectrometers, a macro called "**setlock**" has been created that loads all the lock parameters for the

most common solvents (CDCl_3 , C_6D_6 , CD_2Cl_2 , CD_3COCD_3 , CD_3CN , THF, CD_3OD , DMSO and D_2O ; other solvents will need manual setup.). Issuing this command after choosing your solvent will load the lock parameters as well as the most current shims for the probe and solvent in use. You no longer need to type, for example, "**rts('quad') su**" and manually enter the values for Z_0 , power, etc. from the list taped to the computer monitor. But you still need to check that the lock has been established and fine tune Z_0 if needed. If the lock system didn't engage and the green light is blinking, try increasing *temporarily* the power or the gain. Once the lock is set, turn the power and gain back to their previous values.

One of the most important factors to get a good quality NMR spectrum is shimming. NMR spectroscopy demands a homogeneous magnetic field over the whole of the sample volume located within the probe's detection coil. If the field is not homogeneous the resulting spectra will suffer from distorted lineshape which in turn leads to poor resolution and sensitivity. For this reason, the field has to be optimized to every sample that is introduced in the magnet. The process of adjusting the field's homogeneity is called "shimming". Shimming is achieved with the help of coils (shims) located at strategic places around the sample in the magnet. By applying different electrical currents to these coils it is possible to compensate for deficiencies in the field homogeneity. There can be from 12 to 32 or even more different shims depending on magnet design. These have names related to the axis of the field component they affect the most. For routine operation it is usually necessary to shim only the first two or three Z shims (Z^1 , Z^2 , Z^3).

To shim the magnet you will need an indicator of field homogeneity. This can be provided by the lock signal, whose intensity is proportional to the field's homogeneity. To shim, you make changes to the shims and observe the lock intensity. You continue making changes until a maximum intensity is reached. The actual value is not important, only that you cannot improve it any more. Unfortunately, shimming is not as simple as adjusting each gradient in turn because they interact with each other. That is, changes in one gradient induce a change in another gradient. Fortunately, the interactions are not very large and are well understood. For example, Z^3 interacts mainly with Z^1 and only slightly with Z^2 . There are several procedures for shimming that take into account these interactions. You start by loading a set of standard shims from the computer. These were determined by the Facility staff using a standard sample in a high quality tube. Once loaded, the magnet is pre-shimmed and requires only fine tuning of a subset of all its shims. Here, a very simple procedure for routine applications is described.

The lock signal should be at least 10-20% before starting or shimming will probably be a useless exercise. If it is less than that, check the position of the tube on the spinner and make sure that the lock has indeed "engaged". Turn the lock off, fine tune Z_0 to give a flat, near zero frequency signal, increase the gain to the maximum and turn the lock on again. Also check that the feet of the aluminum ladder is aligned with the blue tape on the floor. The lock signal should go up and the green light (on the meter panel beside the monitor on the Inovax or on the tower adjacent to the magnet on the Mercury) should turn on. Make a small change to Z^1 on the Inova 500 or to $Z^1\text{C}$ on the Inova 400 or the Mercury 300 ($Z^1\text{C}$ is the same as Z^1 , only that the adjustments are Coarse) and observe the lock signal. If the signal increased continue making changes until it starts to decrease. If it decreased, change the shim in the opposite direction. After Z^1 is optimized, proceed in the same manner with Z^2 (or $Z^2\text{C}$). Now, because Z^2 interacts with Z^1 , the latter must be readjusted. Repeat the whole procedure several times until no further improvement is possible. Usually, it is only necessary to adjust Z^1 and Z^2 but you can also adjust Z^3 and Z^4 to get better lineshape. If you want to do it, change Z^3 , then readjust Z^2 and Z^1 as described and repeat until you reach the maximum. Then proceed with Z^4 , change it and readjust Z^1 , Z^2 and Z^3 as described.

Broad, symmetrical peaks indicate that Z^1 and Z^3 need optimization while asymmetrical peaks with long tails or shoulders on either side indicate faulty Z^2 or Z^4 settings. The higher the order of the gradient, the closer to the peak's base its effect will be. Large spinning side bands indicate poor non-spinning shims.

These are shims with components in the X or Y axes and as the name implies, they must be adjusted with the spinning turned off. If you observe large spinning side bands, turn spinning off and adjust X and Y interactively using the procedure for Z^1 - Z^2 .

You can also use *automatic gradient shimming* on the 400 and 500 instruments. This procedure shims Z^1 - Z^6 simultaneously and usually does a decent job. To use it, type "**gradshim**" and wait until it finishes. If your tube requires more than the normal shimming, it may be necessary to run `gradshim` twice. Give it a try, it may even give better results than your manual shimming! At the very least, it will provide a much better starting point to start manual shimming and doesn't even require the sample to be locked.

Keep in mind that some solvents, particularly acetone and methanol, react more slowly to changes in the shims due to their typically long relaxation times. With these solvents shimming has to be done with low lock powers, slowly and patiently.

It is also possible to shim non deuterated solvents on the Inovas. To do it, select the equivalent deuterated solvent from the list, turn the lock off and type "**gradshim('H1')**". Locking is not possible, of course, but this is not a problem with short term acquisitions, like routine 1D spectra, as the magnetic field in modern superconducting magnets is very stable.

After shimming and locking, you can change acquisition parameters either by typing them on the command line of `vnmrj` or you can modify the default values in the *Acquire* panel.

References:

1. T. Claridge. *"High-Resolution NMR Techniques in Organic Chemistry"*. Pergamon Press **1999**, pages 87-94.
2. Varian Inc. "*VnmrJ Liquids NMR User Guide*". Varian Inc. 2003. Section 3.9.
3. V. W. Miner and W. W. Conover. "*The Shimming of High Resolution NMR Magnets. As told by a couple of nuts*". <http://www.acornnmr.com/Sam/shimintro.htm>
4. G. A. Pearson. "*Shimming an NMR Magnet*". <http://nmr.chem.uiowa.edu/manuals/Shimming-GAP-NMR-magnet.pdf>
5. M. M. Abell. "*Mikes Method for Magnet Shimming*". <http://www.technion.ac.il/~balazs/docs/Mike.pdf>
6. K. Brown. "*Magnet shimming*". <http://chem4823.usask.ca/shimming.html>