The Lock

As was stated earlier the magnets used in high resolution NMR are not perfect and are prone to drift for a variety of reasons. To compensate for this drift and to hold the magnetic field as stable as possible the field-frequency lock was developed (in the days of electromagnets this was also known as a super-stabilizer). An example of magnetic field drift can be seen in the following figure:

The lock unit can also be said to be a component of the shim system as the lock signal is the most often used criterion for adjustment of the magnetic field. In short the lock unit is for all intents and purposes a self contained mini-NMR which measures most often the resonance position of deuterium. In choosing deuterium as the lock nucleus several things are accomplished:

1. Deuterated solvents are used which have the same properties of the "light" solvents with the added bonus of having a substantially reduced proton signal (some solvents are > 99.99% deuterated).
2. Deuterium is a spin 1 nucleus and hence has a reasonably short T1 compared to 1H permitting the use of rapid pulse-acquire or swept CW measurement.
3. The deuterium signal can be used as an internal reference as the resonance position of the 2H signal can be compared to other nuclei by using a ratio of the gamma's to assign 0ppm for whatever nucleus you are observing.

Of course this means that the probe (which will be talked about in the next section) must be tuned for deuterium excitation and detection.

A schematic of the lock sub-system is shown below:

The lock transmit/receiver generates the excitation pulse using frequencies supplied by a dedicated reference frequency generator which has been supplied with the absolute lock frequency (the frequency at which you want to keep the field steady at). On the Varian spectrometers this is a parameter called lockfreq and is one of the fundamental console parameters from which many other reference offsets are derived.

The degree to which the deuterium spins are tipped is a function of the parameter lockpower (analogous to the transmitter power on the observe channels). This parameter is quite important for the stability of the lock as too much lock power leads to flip angles which are too large and the signal then becomes erratic. This is known as lock saturation and leads to instability in the magnetic field. Some solvents which have a very sharp deuterium resonance (and hence long T2 and T1) can saturate very easily i.e.: acetone-d6, acetonitrile-d3 etc.

The lock signal coming back from the lock coil is amplified and fed into the lock receiver. The amplification step is controlled by a parameter lockgain which is analogous to the observe preamp gain. In the first lab guidelines for optimization of lockpower and lockgain will be presented. As was talked about in the observe receiver section the lock signal is detected in quadrature (for optimum performance the lock should be exactly on resonance) as the field correction circuit must know whether the lock signal is drifting higher or lower than the reference frequency. For some lock systems a dispersive signal is used which gives equal but opposite intensities on either side of the reference frequency. An integrator sums the components to zero when the lock is directly on resonance. If the sum is not zero a correction circuit sends a small amount of electrical current into a resistive "room

temperature" coil which is part of the room temperature shim tube assembly. The coil is wound in such a way that the current either augments or detracts from the main B0 field in such a way as to return the integrated sum to zero.

A schematic view of the lock field-frequency is shown below:

The lock signal is also digitized and presented to the operator as a "lock level" with which to use as a guide to optimizing the magnetic field.

Establishing a lock

Once the sample is seated in the probe click the SPIN: on choice and see tht the sample spins smoothly up to the default setting of 20 Hz. If your sample was in the same solvent as the previous one you may click the LOCK: on choice and the machine should display the message LOCKED

You may need to adjust the lockpower and or lockgain sliders to position the locklevel to be ca. 70 - 90% To adjust any of these parameters note that the buttons labeled -1+ -4+ etc subtract that value by clicking on the button with the left mouse button and add that value by clicking with the right mouse button. You may also grab the slider with the left mouse button and slide to a new value.

Please note that for CDCl3 the appropriate lock power is ca. 20 and the appropriate lock gain is ca. 20. If your sample is in a different solvent than the previous one you will have to adjust the Z0 offset to that appropriate for your solvent. Since the graphic displays the actual lock NMR signal if you are far away from the Z0 "resonace" postion of your lock solvent the display will appear as in figure 6

As you move the Z0 setting closer to the resonance position for your solvent the oscillations begin to slow and the graphic display looks like this image below:

As Z0 moves to the resonance position the graphic display looks like the image below:

At this point clicking on the LOCK: on choice will engage the lock! The machine will go from displaying LOCK OFF to LOCKED . You are now ready to optimize the lock condition and then go on to shimming.

For Easily Finding the Lock Signal Please Refer to the Following Table of Z0, Lockpower and Lockgain Settings:

Remember that you must adjust the lockpower so as not to saturate the lock signal. This is manifested by a wildly changing lock level and indicates too much lock power. The lock pahse should be realtively constant from sample to sample and should not need adjusting. Please note however, that if the lock phase is 180 degrees away from optimal it will not establish lock. If you have trouble locking the spectrometer please see one of the NMR Center personel.

Current Lock Z0 Settings for October 2001

Solvent	Z0	Lockpower	Lockgain
CDCl3	*474*	*20*	*20*
Benzene	*480*	*16*	*20*
DMSO	*250*	*18*	*20*
Acetone	*225*	*12*	*15*
D2O	*340*	*14*	*20*

Lock tips and exercises

If you are establishing the lock by hand (not using the autolock function) and you are going to shim by hand (not use the autoshim or gradient shim function) then you will want to establish the lock with an eye for maximum stability.

The overall lock stability is dependent on having a good signal to noise ratio. Having a low lock power and a high lock gain may lead to an overall high level but the resulting signal will be quite noisy (the tip of the bar will jump back and forth). On the other hand we have already seen that having a lock power which is too high leads to saturation of the lock signal and field instability. The best approach is to adjust the lock power setting so as to know where the lock begins to saturate (you will probably have to reduce the gain to a fairly low setting) . At this point you bring the lock power down to give a a signal at about 50% of maximum. Using the gain you can now adjust the signal to be between 60 - 80% to begin the shimming process. As you shim the lock level hopefully will go up. As it does you can now use the lock gain exclusively to lower the signal back into the 60-80% range. For complete topping off you can adjust the lock phase to further maximize the signal. For some probes this is important when shifting to samples of different dielectric.

If the lock level goes below 15% then as you begin the shimming process you may find the spectrometer "loses" lock.

Exercises

1. Purposely drive the lock into saturation to see the effects

    a. Lower the gain and increase the power on the CDCl3 sample, record the lock power and gain settings that you find.

    A sample in Acetone-d6 can then be put in the magnet and you can see the difference in the lock power which causes saturation.

2. Turn the lock off and move the Z0 setting around to see the effect of being far off resonance and then coming into resonance.
   At this point we are on to the process of shimming, but first a digression...