

Locking your NMR Sample

This handout contains instructions on how to lock on any deuterated solvent. The Lock on a modern Varian spectrometer is essentially a separate lock-sweep spectrometer that is specific to deuterium. Locking is simply the adjustment of the deuterium lock field (Z_0) such that the deuterated solvent's resonance (the deuterium signal) is centered on a predefined lock frequency. The spectrometer then uses this information in order to compensate for any drifting of the magnetic field (i.e. small loss over time of field strength). Without this drift compensation, your sample peaks would apparently appear at a different frequency over time leading to peak broadening. Modern NMR magnets generally do not drift more than a few Hertz per hour, which is small relative to the overall field (only 0.01 ppm at 500 MHz) but is large enough to wash out J-coupling for an hour-long experiment. Thus, locking is rather necessary. Furthermore, shimming on the lock is typically easier than shimming on the FID.

FINALLY, PLEASE FOLLOW A FEW SIMPLE HOUSE RULES:

1. OBSERVE ALL OPERATIONAL PROCEDURES CAREFULLY.
2. IF THERE IS SOMETHING THAT YOU DON'T KNOW OR ARE NOT SURE ABOUT, PLEASE ASK.
3. USE COMMON SENSE AND DON'T RUSH -- THIS WILL GO A LONG WAY IN AVOIDING COSTLY MISTAKES.
4. REPORT ANY PROBLEM TO **LAB STAFF** AND/OR WRITE IT DOWN IN THE LOGBOOK.

Explanation of Types of Commands Found in this Handout:

1. The VNMR software and the UNIX operating system are both case sensitive. This means that the computer distinguishes whether the letters are entered in upper case (*i.e.* CAPITALS) or lower case. The user must be careful to type the correct case for each letter in a command.

*Example: **jexp1** is not the same as **JEXP1***

2. Some commands are line commands and are typed in by the user followed by a return (a Return is assumed for typed **bold** text commands).

*Example: **su***

3. Some commands are executed by clicking a mouse button with its pointer on a "button" found on the screen. The execution of these commands are indicated by a two letter designation (LC {left click}, RC {right click}, or CC {center click}) followed by a word or words in **shadow** that would appear in the button.

Example: LC Main Menu

This means to click the left mouse button with its pointer on the button that says "Main Menu".

4. Some commands are executed by the mouse itself. These commands are indicated by a two letter designation (LC, RC, or CC) and a description of what the user should do in parentheses.

Example: LC (at 6 ppm)

This means to click the left mouse button with the mouse cursor positioned at 6ppm.

5. Parameters are entered by typing the parameter name followed by a equal sign, the value, and a return.

*Example: **nt=16***

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Insert the Sample

LC **acqi** (top menu bar, right-hand side) connect to VNMR ACQUISITION window
LC **eject** eject sample.

1. *Remove the sample:* Carefully climb the ladder and remove the sample from the top of the upper barrel of the magnet. *Grab the fat part of the spinner and be sure to avoid touching the narrow part of the spinner to keep it clean and prevent spinning problems.*
2. *Position your sample in the spinner:* Remove the standard from the spinner. Insert your sample in the spinner and use the depth gauge sitting on the console to position the sample. Make sure that the solvent is centered about the two white lines (etched in the gauge). *When positioning the sample, make sure that your eyes are at the same level as the bottom of the tube in the gauge.* Otherwise, the sample could be incorrectly positioned by as much as 5 mm. Ask the LAB staff or your trainer for a demonstration of this the first time you train on this instrument. If you do not position the NMR tube to within 1 mm of the position on the gauge, the standard shim libraries may not be very close and a substantial amount of shimming may be required. If you position the NMR tube lower than allowed by the gauge, the sample will not spin.
3. *Place the sample:* Place your sample and spinner on the top of the upper barrel of the **magnet (with the eject air still on)** and make sure that the spinner does not get caught on the lip of the spin stack. (If this happens, the sample will hang momentarily when the eject air goes off, then fall suddenly and usually break in the probe when it hits the bottom.) Then,

LC **insert** insert sample (listen for two clicking sounds as the eject air goes off and the sample slowly drops in).

Lock on the Sample

Still in the VNMR Acquisition Window,

LC **LOCK** connect to interactive lock window.

NOTE: Next to the **LOCK** button at the top of this window is a button labeled **SPIN:** with off/on buttons next to it. The **SPIN: on** button should be highlighted. The spin rate is set at the bottom of this window with the Spin button. The rate should be set to 20: if it isn't, please set it to 20 at this time. The current spin rate is displayed at the bottom of the lock window.

Wait for the lock signal in the lock window to appear, then:

LC **LOCK:off** turn lock off.

THE FOLLOWING IS A SHORTCUT. IF YOU ARE SWITCHING SAMPLES THAT HAVE THE SAME LOCK SOLVENT, YOU SHOULD NOT HAVE TO OPTIMIZE Z0, LOCKPOWER, LOCKGAIN, OR LOCKPHASE.

Simply,
LC LOCK:on turn the lock on.

IF THIS DOESN'T RESULT IN A LOCKED CONDITION INDICATED BY A SINGLE STEP IN THE LOCK LEVEL, GO THROUGH THE PROCEDURE BELOW. REMEMBER, IF YOU ARE CHANGING SOLVENTS, YOU MUST RESET Z0, LOCKPOWER, LOCKGAIN, AND LOCKPHASE.

NOTE: If you click on **LOCK:off** before the lock signal appears, the lock status may be incorrect, *i.e.*, the lock could be off while the indicator is at the "**LOCK:on**" position, and *vice versa*. If this happens, close and reconnect to VNMR ACQUISITION window.

Adjustment of Parameters in the Interactive VNMR ACQUISITION Window

Place the mouse pointer on a button (such as -1+) for the appropriate function (such as **lockpower**) and *left click* to *decrease* its value by the number of units selected, or *right click* to *increase* its value by the same amount. An alternative is to left click and drag the slider in either direction; however, be careful not to move the slider too fast as you can easily miss the on resonance condition.

Remember, the lock must be in the off condition to change Z0:

LC LOCK:off turn lock off.
RC lockpower and **lockgain** buttons increase lock power (~50) and lock gain (55 - 60).

NOTE: In general, a lock power greater than the normal setting is used for locking (10-30 units). With increased lock power and maximum gain, a sine wave should be seen if the lock is off resonance. If the lock is on or near resonance, a flat or near flat step line will be seen which may rise up under these conditions.

LC or **RCZ0** buttons "zero beat" the sine wave to bring the lock to resonance condition.

This means that Z0 is adjusted until the sine wave becomes a flat line with a step, which is an indication that the lock is on resonance. Then:

LC LOCK:on turn the lock on.
LC lockpower buttons reduce lock power. In general, this is set to around 20-25 for most CDCl₃ samples, 10 - 15 for C₆D₆, and 5 - 10 for acetone-*d*₆. The lock level should not significantly fluctuate if the power is set correctly.
LC lockgain buttons adjust lock gain to maintain a lock level of 40-90%.

ALWAYS MINIMIZE LOCKPOWER, MAXIMIZE LOCKGAIN!

LC or **RC lockphase** buttons adjust lock phase to maximize the lock level.

NOTE: Lock phase is dependent on the shims. After its initial optimization, one should readjust the lock phase after shimming on Z1C and Z2C, especially when large changes of the shim gradients have been made. Shimming on the lock level requires that the lock phase be adjusted correctly.