

Locking on the U400

Once you have logged onto the spectrometer *via* Reslog and inserted the sample (see handout UVU405, *U400 Sample Preparation, Positioning, and Insertion*),

1. Change Current Working Directory to Your fid Directory

name <rtn> enter your username at the prompt
mydir <rtn> change working directory to your fid directory

2. Retrieve a Standard Shim File for a Given Probe and Solvent

rts('filename') <rtn> see Table 1 for a list of shim files for the QUAD probe

3. Select a Set of Standard Parameters for a Given Nucleus and Solvent

LC Main Menu select main menu
LC 2:Setup select setup menu
LC 1:H1,CDC13 (for example) select standard parameter for ¹H nucleus in CDCl₃
dg <rtn> display acquisition and processing parameters and make necessary changes.

4. Perform an Experiment Setup

su <rtn> perform experiment setup
load='n' <rtn> set load shim values from current experiment to "no"

5. LOCK ON THE SAMPLE

LC Connect (in VNMR ACQUISITION window) connect to VNMR ACQUISITION window
LC LOCK connect to interactive lock window
LC LOCK:off turn the lock off

Adjustment of parameters in the interactive VNMR ACQUISITION window:

Place the mouse pointer in 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.

RC the **lockpower** and **lockgain** buttons increase lock power and lock gain until a sine wave is seen if the lock is off resonance. If the lock is on resonance, a flat line will be seen which may rise with the increased lock power and gain.
 LC or RC the **Z0** 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 and rises up, which is an indication that the lock is on resonance. Then

LC LOCK:on turn the lock on
LC lockpower buttons adjust lock power, which is solvent and concentration dependent. See Table 1 for examples.
LC lockgain buttons adjust lock gain to maintain a lock level between 40-90. Lock gain is normally set to between 40 and 55, depending on the solvent and sample concentration.
 LC or RC **lockphase** buttons adjust lock phase to maximize the lock level

NOTES:

- 1) Concentrated samples may require higher LOCK POWER and LOCK GAIN.
- 2) LOCK PHASE is dependent on the shims. Therefore, after its initial optimization, one should readjust the lock phase from time to time during the shimming process, especially when large changes of the shim gradients have been made. Shimming on the lock level requires that the lock phase is adjusted correctly.
- 3) Some NMR solvents, such as CD₃OD, C₆D₅CD₃, THF-*d*₄, and pyridine-*d*₅, have more than one deuterium lock signals. If the strength and sharpness of these signals are different, one should lock onto the stronger (*i.e.*, the one with a higher number of EQUIVALENT deuteriums) and sharper signal, such as the methyl signal in both CD₃OD and C₆D₅CD₃. THF and pyridine are trickier to lock onto due to the fact that the lock signals in these solvents not only have similar strength and sharpness, but also have similar chemical shifts (3.58 and 1.73 for THF and 8.71, 7.55, and 7.19 for pyridine).
- 4) When the Z0 of a solvent is unknown, one can calculate its approximate value from the chemical shift of its residue 1H NMR signal (δ): $Z0 = -23 + 61.4(7.26 - \delta)$. (-23 is the current Z0 for CDCl₃ which may change with time, 7.26 is the chemical shift of the residue CHCl₃ signal, and 61.4 is the deuterium frequency at 9.39 Tesla, the field strength of the U400.) For example, Z0(Ac-*d*₆) = -23 + 61.4(7.26 - 2.04) = 298. This method can also be used in the case of one solvent having more than one lock signals to make sure that one has locked onto the correct signal by checking the experimental Z0 against the calculated one.
- 5) When an appropriate set of standard parameters for the solvent has been selected and the correct deuterium signal has been locked onto, the reference for the spectrum should be very close to where it should be. This could be of some importance in situations where the solvent signal can not be easily identified or can not be identified at all due to overlapping. If you have locked onto the wrong deuterium signal, such as locking onto the aromatic signals (7 ppm) in toluene instead of the methyl signal (2.09ppm), the reference for the spectrum will be off by 7 - 2.09 = 4.9 ppm and your spectral window will be shifted to 20 to 0 ppm instead of the normal 15 to -5 ppm.
- 6) In addition to the solvents contained in the 3:Nucleus,Solvent menu, which includes CDCl₃, D₂O, Benzene, DMSO, and Acetone, the following solvents are also supported in the 6:Other sub-menu (be sure to enter the name or formula EXACTLY as shown below):

CD ₃ CN	CD ₂ Cl ₂	Methanol / CD ₃ OD (3.30)
Toluene / C ₆ D ₅ CD ₃ (2.09)	AceticAcid / CD ₃ COOD (2.03)	THF / thf (3.58)
Pyridine / C ₅ D ₅ N (7.19)	Cyclohexane / C ₆ D ₁₂	DMF (2.91)

The chemical shift in the parentheses indicates the correct deuterium signal that you should lock onto for that particular solvent. If you are using a solvent that is not listed above, you can select a solvent from the above list that has the closest chemical shift to the deuterium signal that you are locking onto, or you can provide me with the chemical shift information of that solvent and I will add it to the existing list.

Table 1. Locking Parameters and Standard Shim Files for the QUAD Probe (11/12/93)

SOLVENT	CDCl ₃	C ₆ D ₆	D ₂ O	CD ₃ OD	Ac- <i>d</i> ₆
Z0*	-23	-16	136	220	298
LOCK POWER	20 - 25	10 - 15	10	5 - 10	5 - 10
SHIM FILE	QUAD	QUAD	QUADD2O	QUADMET	QUADAc ₆

* These are the current Z0 values, which will change with time.