VI. **Trouble-Shooting**

 wrong parameters - make sure **probe** parameter matches probe in magnet

 sample won't spin - if probe has been changed, find TA to try reseating spin collar tube: push probe up, then at top of magnet, push down the

aluminum tube guiding sample in

- check that sample tube is not inserted too far into spin collar

- check that VT air is not turned up too high

- try turning the VT air flow up to (turn it back down after • sample won't eject

inserting)

- try "air" hook up (rather than N₂ gas which has lower pressure)

- check that air pressure (gauge in southeast corner) is turned up to

mark

• sample won't shim - read in proper shim file (use UWMACROS LOADSHIMS)

> - check that you have enough solvent (≥0.6 ml) and are 67mm down on depth gauge, and centered in rf region

- check that lock power is not too high, and that lock phase is correctly adjusted

- let magnet warm for quite a while (up to 1h) after a cold

experiment

- if previous student didn't stop early enough, you will need to adjust especially the lock phase fairly often during the warmup (and wack the previous person as hard as possible with a wet

noodle!), and also reshim somewhat over 30 min to 1 hour

• command doesn't work – hit return key and try again; some mistypes carry over to next

line

• S/N seems poor - most likely, an attenuator has been left in line at the output of the

> preamp leading back to the ADC; if your sample is not very concentrated, remove this attenuator and adjust the gain setting

- check pw90 (at least on the observe side); if unusually long,

check with TA or facility staff

spectrum on screen is only an inch long or so

- type **full** to reset plot window (needed after **dssh** command)

- enter the command unlock(#) where # is the exp number that's says exp locked

locked, or delete the file ~/vnmrsys/lock #.primary in unix

won't let jexp# - probably have not created the experiment (see WORKSPACE)

> will list all experiment areas explib cexp(#) will create experiment area #

will delete experiment area # (saves disk space) delexp(#)

- cannot get good pw90 calibration
 - check that probe is properly tuned
 - check that **tpwr** is set correctly (or **pwxlvl** for decoupler calibration)
 - check that external attenuator is *not* placed in ¹H observe position
- waits a long time before acquisition starts have one of the following flags set

spin ≠ 0
 if spin is set to a number, the spectrometer will "regulate" the spinning,
 taking time before acquisition to make sure the spinning is regulated;
 set spin=0 at the vnmr prompt, and set spinning in the acqi window
 for this setting, spectrometer will perform an autogain; recommend
 setting the gain to a specific value manually and not using autogain
 autoshimming will occur; should not be used except possibly between
 kinetic runs (simply too inefficient and wastes spectrometer time)

- use the **flagsoff** macro to set all the flags above to their appropriate values
- No acqi (lock-n-shim) window: Type acqi in vnmr command line
- Can't Connect to spectrometer
 - Pressing connect button on *acqi* window doesn't work:
 Try in UNIX terminal window: su acqproc twice (once to kill, once to restart); this should re-enable connect to *acqi*
- **FIFO Underflow Error:** Check sweep width; an excessive sweep width (>80,000 Hz) can show this error, try reducing sw and re-acquiring.
- **loc not defined:** Type the macro **fixloc** to correct.