V. LOCKING ON THE DEUTERATED REFERENCE SOLVENT

A) Introduction to the Lock and Lock Display

Before trying to lock or shim, one should always load a shim library that is appropriate for the solvent and tube size being used. If there isn't a shim library specifically for your sample, use one that would correspond most closely in a proton spectrum to your solvent.

To load a shim library, type \textbf{AH} and the single letter sub COMMAND: \textbf{L}. This will display the available shim libraries, one at a time. Press \textbf{return} until the name of the desired shims is found. Type \textbf{L} to "load" this desired set of shims. Type \textbf{X} to exit the \textbf{AH} subroutine.

The QE300 locks on the deuterium signal in the solvent. More deuterons per volume will increase the ability to obtain a strong lock easily. Thus, 100% deuterated solvents are preferable. CDCl$_3$ is less easy to lock onto than most deuterated solvents, but it works well enough and is less expensive. If you did not enter the correct lock frequency (in PPM) upon lowering the sample into the probe, this can be done in \textbf{LD} by typing \textbf{F} (for frequency) and \textbf{M} (for manual entry) and entering the lock frequency (in PPM) for your solvent and depressing \textbf{return}. (Refer to handout #033 for the proper values of common lock solvents. Use the ppm value that the solvent would show up at in a proton spectrum.)

If the Lock Frequency is properly set for the lock signal to be within range, and if a shim library appropriate for the sample has been entered, the lock will often reestablish itself as soon as the sample is in place in the probe. Occasionally, with weak lock signals, it may be necessary to increase the Lock Gain and/or Power briefly, in order to establish a locked condition.

The lock level meter is displayed numerically on the top line of the monitor, toward the right, or as a bar graph in \textbf{LD}. One should not consider the sample locked on $^2$H unless it is showing a lock level of greater than 30 units. Normally the lock level should be about 80. When shimming, the greatest sensitivity is observed at 90 to 95 units. Use the Lock Gain to adjust it to these levels.

The \textbf{LD} routine can provide either a lock level bar graph display or a lock dispersion signal display as the field is continually swept (see the figures below). The \textbf{S} subcommand of \textbf{LD} toggles between the two displays, unless an acquisition is in progress.

\begin{center}
\textbf{LOCK LEVEL METER BAR GRAPH DISPLAY}
\end{center}

\begin{center}
\begin{tabular}{c c c c c c c}
FREQ(PPM) & PHASE & OFFSET & GAIN(RX) & TXRF LEVEL & WIDTH & DWELL \\
7.15 & 230 & 700 & 741 & 100 & 512 & 256 \\
\end{tabular}
\end{center}
LOCK DISPERSION DISPLAY

Below is a summary of the most useful >LD subcommands:

- **F**: assign knob to change lock frequency
- **P**: assign knob to change lock phase
- **O**: assign knob to change lock offset
- **G**: assign knob to change lock gain
- **T**: assign knob to change lock transmitter power
- **W**: assign knob to change lock sweep width
- **D**: assign knob to change lock dwell time
- **S**: toggle between sweep display and lock level (meter) display
- **L**: cycle lock unit through standby, lock fast, and lock slow
- **K**: enable knob shimming (see section VI on shimming)

<return>  exit lock display routine; return to command mode

These subcommands are active without pressing <return>. All of the entries below the lock level display are accessed by typing their first letter. This function is then assigned to the knob. The function currently controlled by the knob is always highlighted on the screen. One can also manually enter a desired value using the keyboard by typing the first letter of these functions followed by an M and the numerical value. The S subcommand toggles between the lock level and lock dispersion modes, unless an acquisition is in progress.

B) **Explanation of Lock Parameters**

1) **Offset and frequency**: These two parameters interact to allow you to center the lock signal. Set the FREQ value to a value appropriate for your solvent (in ppm), then change the OFFSET to center the lock signal on the display.

2) **Sweep width**: the WIDTH describes the range of the sweep window. As you decrease the WIDTH, you are narrowing in on a specific region of the spectrum. Typically the WIDTH is set to about 512. If you use a solvent which gives a weak signal such as CDCl₃, you may need to decrease the WIDTH to maximize signal strength. Remember, when searching for a ²H signal with a large sweep width, you will have to set the TXRF level much higher (300-400), and then lower this level as you decrease the WIDTH.

3) **Dwell**: The DWELL determines how fast the signal sweeps the display and is normally set to 256.

4) **Transmitter (TXRF) level and gain**: When >LD is entered, the vertical scale (VS) is automatically reset to 12. This allows for proper scaling of the lock signal.
The TXRF level is directly proportional to the power (the signal is increased as you increase the numerical value of the TXRF level). To prevent signal saturation during lock, turn the GAIN to about 700-800 and then set the TXFR power so the signal is on scale and not "clipped". The TXRF level can be set to 175 for most solvents if the recommended concentrations are used. An exception is acetone, for which the level should be set to 100. The TXRF level should not need to be more than 200 when locked, except for very concentrated samples (i.e., 57% menthol) which have few deuterons per volume to lock on to. The gain will usually be in the range of 600-1200. Remember that you should check the TXRF levels after shimming if the lock is unstable. Too much power will saturate the signal.

5) Phasing: Optimizing the phase of the deuterium signal is very important for a proper lock and shim and can be done by entering the PHASE subcommand. If the deuterium signal is not correctly phased, the lock will be unstable. It is important to note the direction of the signal as it sweeps across the screen. The deuterium signal should always deflect up and then down as the signal rings through.

The easiest way to determine the correct phase is to open the WIDTH until you can see some baseline on each side of the signal, as in the examples below. Depressing the space bar will now give two superimposed signals: the normal signal which sweeps to the right and another which sweeps to the left. A signal which is phased correctly will have superimposed baselines (region 1 in the following figures). It will also show equal heights for the initial upward sweep of each signal (region 2).
The single sweep display can be selected by depressing the space bar again.

6) **Gain.** The gain will usually be in the range of 600-1200. It can be increased momentarily on the bar graph display until a lock is obtained and then reduced to bring the lock level to about 80%.

C) **Manual Locking Procedure**

a) Type >**LD**.
b) Check that the correct lock frequency for your solvent has been entered under FREQ.(ppm). If necessary, type **F** to change it with the knob or **F** and then **M** and type in the desired value followed by a **<return>**.
c) Type **S** to get the lock dispersion display (only necessary if the lock level meter bar graph display is showing.) Then press the **<spacebar>** to toggle the bidirectional sweep on. If a rapidly moving expanded display appears, type **<return>**, then **^F**, and then go back into >**LD**.
d) If the lock signal does not appear on the screen, type **W** and use the knob to increase the width (about 2000 is maximum width) until the signal appears. (Or, it may be necessary to increase the transmitter power; see step e.)
e) Type **T** and use the knob to set the transmitter power to ~200 for all solvents except acetone, which should be set to ~100. (In rare instances of dilute solvents it may be necessary to set the transmitter to ~350 in order to find the signal). If the signal appears "clipped" (top and bottom cut off), the transmitter power level is set too high.
f) Type **O** and use the knob to center the signal.
g) Type **P** and use the knob to phase the signal (see section B for details).
h) Type **W** and use the knob to decrease the width to 512 or type **W**, then **M**, then 512 followed by **<return>**.
i) Type **O** and use the knob to center the signal.
j) Press the **<spacebar>** to toggle the bidirectional sweep off and type **S** to get the lock level display.
k) Type **L** until a LOCKED FAST message appears under the bar graph. **L** cycles the lock through three modes - STANDBY, LOCKED FAST, LOCKED SLOW. When the lock dispersion signal is displayed, the lock is in the STANDBY mode. The LOCKED FAST mode is used for shimming and data acquisition, and LOCKED SLOW is used for data acquisition that requires extreme stability.)
l) Type **G** and use the knob to adjust the gain to bring the lock level to ~80.

D) **LOCK PROBLEMS**

If you cannot find the deuterium signal, set the **FREQ** at the proper ppm value for your solvent, turn the **TXRF** level to 400 and increase the **WIDTH** to 2000. If the signal is still not displayed, vary the offset several hundred units. When the signal is found, optimize the remaining parameters.

If the lock is very unstable, try hand shimming Z1 and Z2 with the **K** subcommand and then reoptimize the lock parameters.