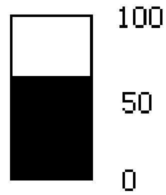
# GN500 Locking

## Introduction

If the correct lock frequency was not entered when the sample was inserted, type **OL**, enter the correct lock frequency for the sample solvent and then **< >**. A deuterated solvent chart is attached to this handout and is also found under the glass on the console. If the solvent has more than one deuterated site, the user should select the sharper peak to lock on (rule-of-thumb is to select the peak with less multiplicity).

The lock level meter is on the front of the console to the left of knob A. When the instrument is locked on the sample solvent, the meter should show an upward deflection. The sample is not locked unless the deflection is greater than 30 units. Normally data acquisition or autoshimming requires that the lock level be +80 units. When hand shimming, the greatest sensitivity is observed between 90 and 95 units.

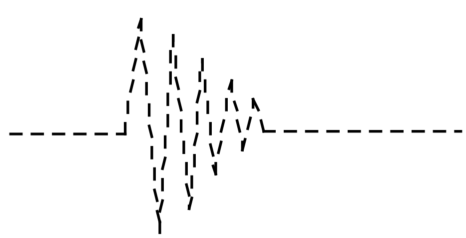
The **LD** routine will display either an image of the lock level meter (as on the console) or an image of the lock dispersion signal as the field is continually swept (see Figure 3). The lock level meter on the console is more accurate than the lock level image on the screen since there are no computer-generated delays and the console meter displays lock fluctuations in real time.If the sweep signal cannot be seen, exit **LD**, take a single scan using **ZG**, and check **LD** again.




STANDBY (or LOCKED FAST, LOCKED SLOW)

| FREQ(PPM) | PHASE | OFFSET | GAIN | TXRF LEVEL | WIDTH | DWELL |
|---|---|---|---|---|---|---|
| 7.26 | 350 | 1350 | 650 | 120 | 512 | 256 |

Figure 3A. Lock level meter display



| FREQ(PPM) | PHASE | OFFSET | GAIN | TXRF LEVEL | WIDTH | DWELL |
|---|---|---|---|---|---|---|
| 7.26 | 350 | 1350 | 650 | 120 | 512 | 256 |

Figure 3B Lock dispersion display

Once in **LD**, several commands are useful to optimize the lock level and lock parameters:

| | | |
|---|---|---|
| **F** | - | assign knob A to change lock frequency |
| **P** | - | assign knob A to change lock phase |
| **O** | - | assign knob A to change lock offset |
| **G** | - | assign knob A to change lock gain |
| **T** | - | assign knob A to change lock transmitter power |
| **W** | - | assign knob A to change lock sweep width |
| **D** | - | assign knob A 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 |
| **A** | - | control spin rate (identical to **AA** command) |
| **< >** | - | exit lock display routine & return to command mode |
| **M** | - | allows manual typed entry of highlighted parameter |

These subcommands are active without pressing < >. All of the parameters below the lock level display are accessed by typing the first letter of the parameter. This parameter is then assigned to knob A and the parameter is highlighted on the screen. The S subcommand toggles between the lock level and lock dispersion modes unless an acquisition is in progress.

Optimizing lock parameters

1. Offset and frequency: These two parameters interact to allow centering of the lock signal. Set the FREQ value to the lock frequency of the deuterated solvent and 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 the WIDTH is decreased, the spectral window is narrowed. Normally the WIDTH is set to 512. If the solvent gives a weak lock signal (such as $CDCl_3$), the WIDTH may have to be decreased to optimize the lock signal strength. If searching for a deuterium signal with a large sweep width, the TXRF level may need to be set higher (200 - 400), and then lowered as the WIDTH is decreased.
3. Dwell: The DWELL determines how fast the signal sweeps the display and is normally set to 256. If the lock signal is saturating even at low TXRF power, increasing the DWELL might help since it will allow more relaxation between sweeps.
4. Transmitter level and gain: When LD is entered, the vertical scale (VS) is automatically set to 12, and allows for proper scaling of the lock signal. The TXRF level is directly proportional to the lock transmitter power. To prevent saturation of the lock signal , turn the GAIN to about 700 - 800 and then set the TXRF power so that the signal is on scale and not clipped (see Figure 4). The TXRF can be set to 90 - 100 for most solvents if the samples are of normal concentration. An exception is acetone and the recommended TXRF power level is 50 - 60. The TXRF power level should never exceed 130 when locked and the gain will usually be between 600 - 1200. Remember that the lock level parameters should be rechecked after shimming, particularly if the lock level has increased dramatically with shimming. Many times both the TXRF power and the GAIN can be decreased after shimming. To check for saturation, turn TXRF power down slightly. If the lock level slowly goes up, the lock signal is probably saturated. Slowly keep turning the TXRF power down until the lock signal does not go up any more. Increase GAIN and continue to drop the TXRF power until it stabilizes.

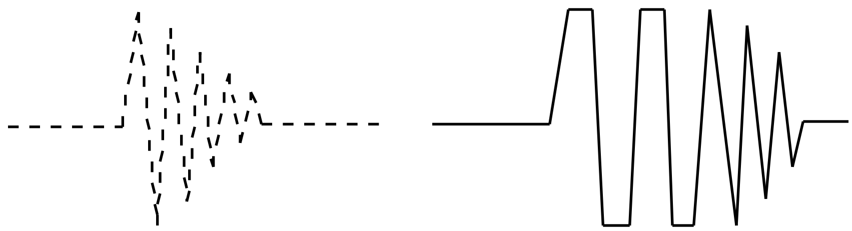


Figure 4. A normal lock signal (left) and a clipped lock signal (right)

5. <u>Phasing</u>: Optimizing the phase of the deuterium signal is very important for a proper and stable lock. If the deuterium signal is not correctly phased, the lock will be unstable. It is important that the lock signal always deflect up first and then down as it sweeps across the screen (see Figure 5). To determine the correct phase, depress the spacebar. Two superimposed signals will appear on the screen. Adjust the PHASE until the images are symmetrical and the baselines are superimposable. The height of the initial upward sweeps should be the same. If it is impossible to get the signals the same height and the height changes from sweep to sweep, check for saturation of the signal. To return to the single sweep signal, press the spacebar again.

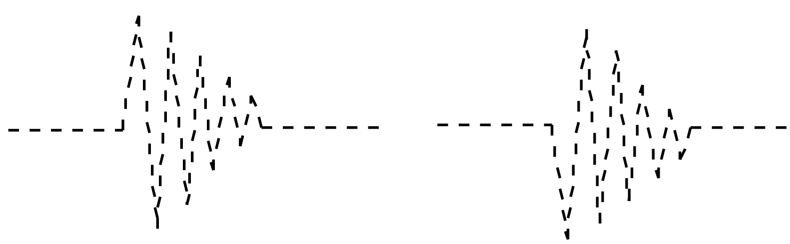


Figure 5. A correctly phased signal (left) and a signal 180° out of phase (right)

## Sample lock procedure

1. Type **LD**.
2. Type **S** to display the lock dispersion signal (only necessary if the lock level meter display is shown)
3. Type **W** and increase the WIDTH to 700 with knob A.
4. Type **F** and enter the correct lock frequency (if necessary).
5. Type **O** and center the lock signal.
6. Type **T** and set TXRF power to 90 - 100.
7. Type **G** and adjust the GAIN until the sweep signal almost fills the screen.
8. Type **P**, then **spacebar**, and phase the signal. Depress **spacebar** when done.

9. Type **W** and decrease the WIDTH to 512.  Check for saturation.

10. Type **S**  to display the lock level meter.

11. Type **L** until the LOCKED SLOW message is displayed under the bar graph.  The LOCKED SLOW mode is used for data acquisition and shimming in the SK mode (**AH**, **K** will only allow shimming in the LOCK FAST mode).

12. Type **G** and adjust the GAIN until lock level is over 80.

## Common lock problems

1. If you cannot find the deuterium signal, set the FREQ at the proper ppm value for your solvent.  Then set the TXRF power level to 300 and increase the width to 1200.  If the signal is still not visible, vary the OFFSET several hundred units in each direction.  When the signal is found readjust the lock parameters.

2. If the lock is very unstable, try hand shimming Z1 and Z2 with the **K** subcommand.  Once the lock level improves, re-optimize the lock parameters to compensate.

3. If no sweep signal is visible, exit the **LD** routine with **< >** and take a scan (**ZG**).  (For this, it does not matter if GEM or GEM16 is loaded.)  Return to **LD** and see if the sweep signal has appeared.  It might be necessary to go through a software reload if the sweep signal is not visible.  The reload procedure is under the glass on the console and given below:

>**MO**<br>
**>XM**, **#2**, **Y**<br>
**>ZG** (It is necessary to take one scan in GEM to initialize the dwell clock)<br>
>**MO**<br>
.**GEM16**<br>
**< >**<br>
**>XM**, execute the appropriate $^{1}$H setup macro # for the probe and nucleus desired<br>
**>ZG**

Check and make sure that an appropriate shim library is still loaded.

4. Be sure that the sample contains enough deuterated solvent to give a lock signal.