2/11/2011

Detailed locking procedure for Varian Spectrometers using VNMRJ

The lock circuit regulates the main magnetic field at a constant strength. It works by monitoring the deuterium signal of the solvent and continuously comparing it to an internal reference signal. Variations in the superconducting magnet change the resonance frequency of the lock signal. The lock circuit detects that change as a frequency difference between the lock signal and the internal reference and cancels variations in the field by adjusting the current in an electromagnet called **Z0** so that the effective magnetic field remains constant ($B_{eff} = B_{supercon} + B_{Z0}$).

For the lock circuit to operate correctly you must 1) match the frequency of the lock signal to the frequency of internal reference signal by adjusting **Z0**; 2) match the amplitude of the lock signal to the amplitude of internal reference signal by adjusting the **lock** transmitter **power** and **lock** receiver **gain**; and 3) match the phase of the lock signal to the phase of internal reference signal by adjusting the **lock** receiver **phase**.

Schematic of the Lock circuit
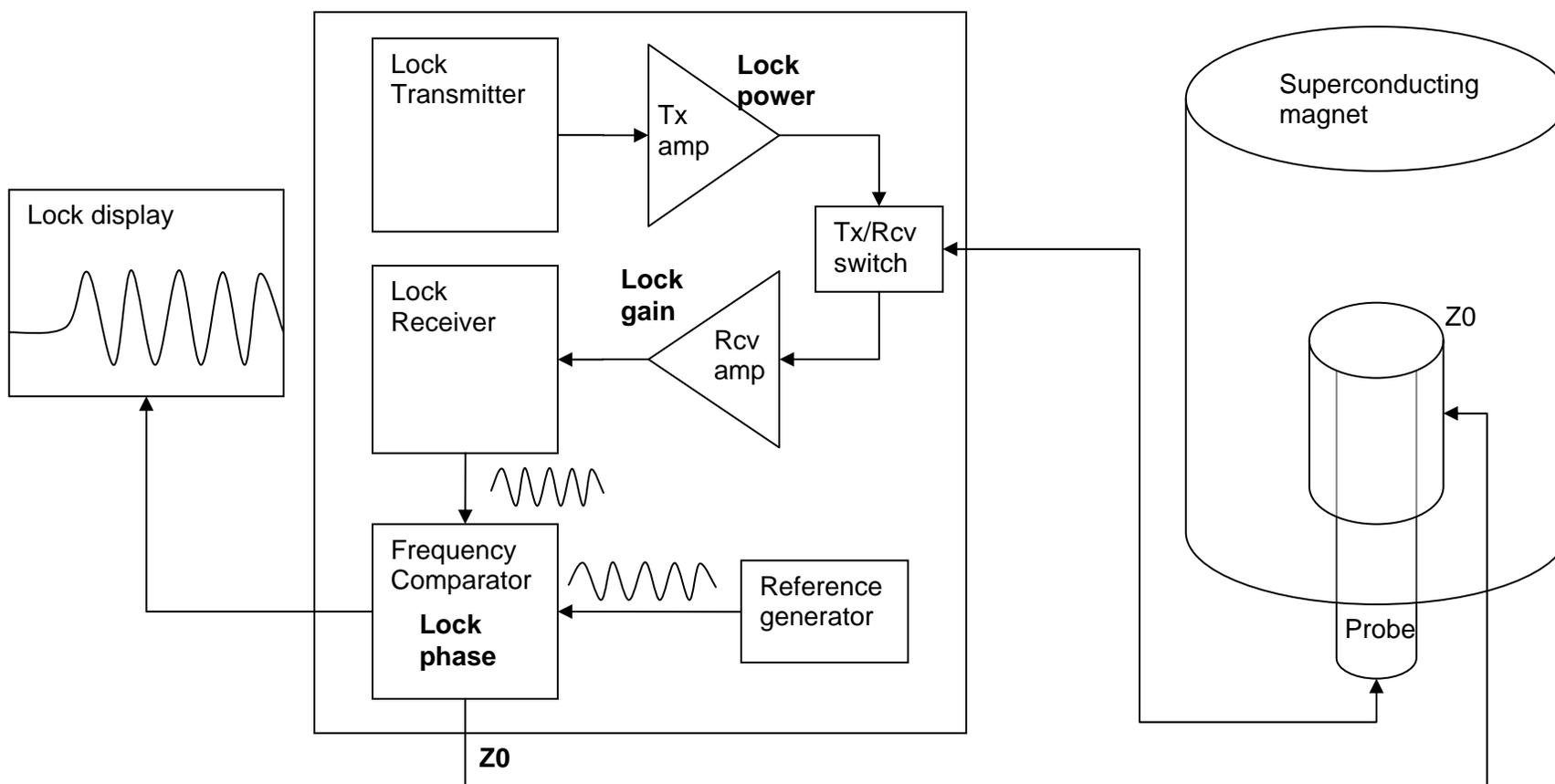
Automatic Locking/Shimming

1. Click Start→Standard

2. Select the correct solvent (this is essential for the automation routines)

3. Click "Find z0"

4. Wait for "Find z0" to finish. Don't worry about the lock status at this point.

5. Click "Gradient Autoshim"

6. Click Start→Lock and confirm that Status under the Lock ON/OFF buttons in the top left corner of the parameter panel reads "Regulated"

7. If the status reads "Not Reg" follow the manual locking instructions below or for short acquisitions (minutes) you can turn lock OFF and run your sample unlocked. Do not run experiments with the lock not regulated.

NOTE: The solvent selection will change to D_2O during Find Z0 and gradient autoshimming. This is normal. Your selection will be restored when gradient shimming is complete. If the software crashes during Find Z0 or gradient shimming you may have to reselect the solvent manually.
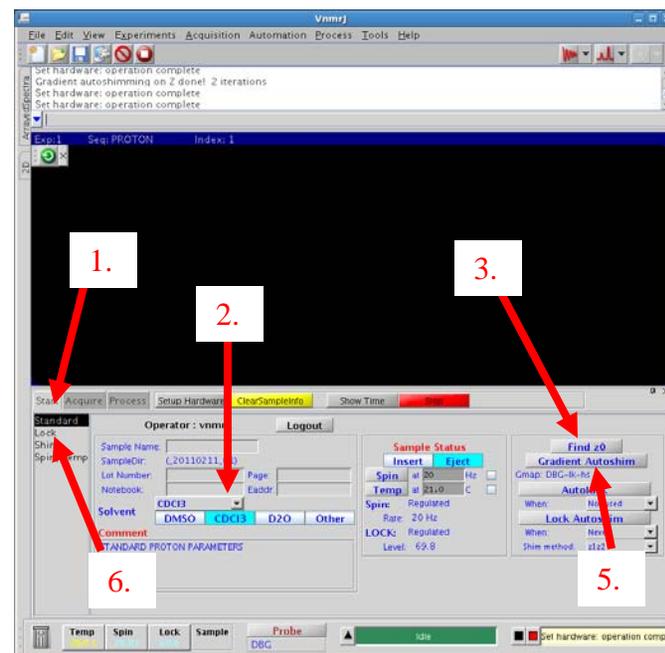
Manual Locking

1. Click Start→Lock

2. Click Lock OFF

3. Click Lock Scan

4. Increase Lock Gain by dragging the slider and

5. Increase Lock Power by dragging the slider until

6. you can clearly see a sine wave in the spectrum display window.

NOTE: For chloroform you will likely need to maximize Power and Gain. Solvents like acetone-d_6, methanol-d_4, DMSO-d_6, D2O, and benzene-d_6 have strong ^2H signals relative to $CDCl_3$ and so you may need to use lower lock power and gain settings to keep the signal on screen.
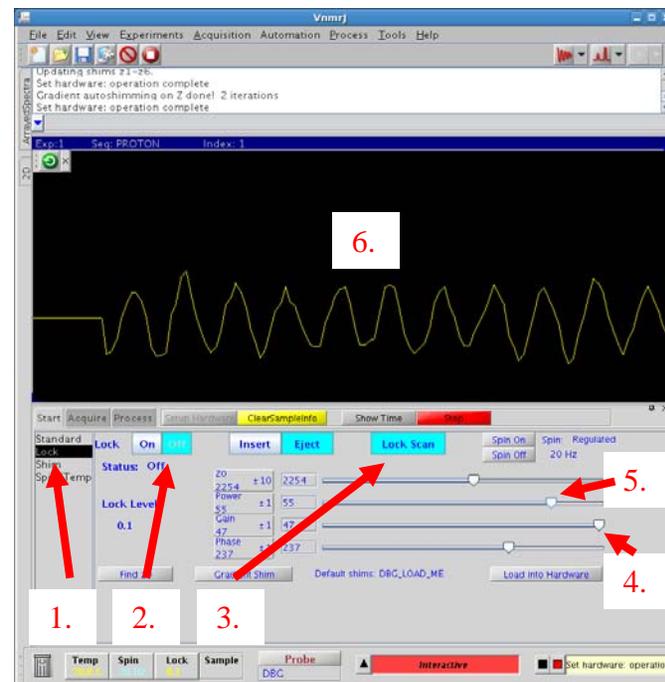
7. Observe the lock signal.

    a. If the signal looks like a "step function", or "0 frequency sine wave" (see Figure 3), skip to step 11.

    b. If it looks like a low frequency sine wave (Figure 2), skip to step 10.

    c. If it looks like a high frequency sine wave (Figure 1), or you don't see a sine wave at all continue to next step.

8. Change **Z0** by dragging the slider ~½ inch (~1 cm) in either direction and **wait 3 seconds** for the signal to stabilize.

    a. If the sine wave decreases in frequency (that is, you see fewer oscillations of the wave on the screen), you are going in the correct direction and you should continue moving the slider in the same direction.

    b. If the sine wave increases in frequency (that is, you see more oscillations of the wave on the screen), you are going in the wrong direction and you should move the slider the other way.

9. Continue adjusting the **Z0** slider until you see a low frequency sine wave (Figure 2). **NOTE:** Concentrate on the frequency of the signal and ignore intensity fluctuations—intensity is meaningless at this point.

10. Use the **Z0** button to make progressively finer adjustments until the signal flattens out and looks like a "step function" (Figure 3). The sensitivity of the button is adjusted by **clicking** (not scrolling) the mouse wheel. Use single clicks in +/- 100 mode or click and hold in +/- 10 mode.

11. Maximize the Lock Level using the **Z0** button with single clicks in +/- 10 mode or click and hold in +/- 1 mode. You may need to reduce lock power to keep the signal on screen or if the lock level "breathes" (drifts up and down on its own). If the signal gets noisy, reduce lock gain instead of power.
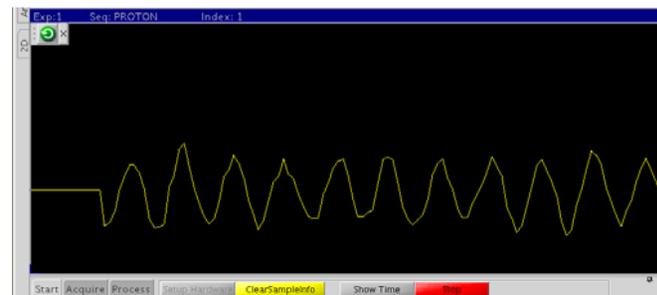

Figure 1 High frequency sine wave: Z0 far from correct setting. Minimize frequency using the Z0 slider.

Figure 2 Low frequency sine wave: Z0 close to correct setting. Minimize frequency by clicking the Z0 button in +/- 100 mode or clicking and holding button in +/- 10 mode.
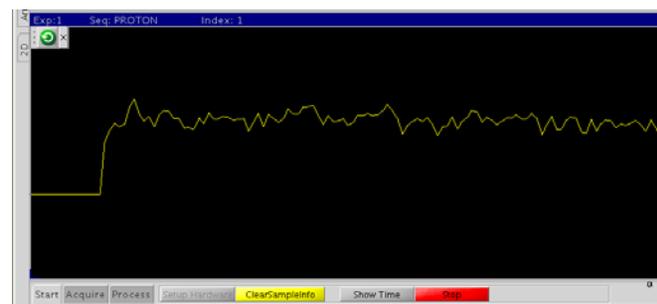

Figure 3 Zero frequency sine wave or "step function": Z0 at correct setting. Maximize intensity using the z0 button in +/- 10 or +/- 1 mode. If signal goes off scale (lock level > 100) you should reduce lock power.

12. Turn on the lock. You should see "Regulated" appear below the lock ON/OFF buttons. Also, the "Lock" light on the remote status display should be lit (not blinking).

13. Adjust **lock power** so the lock level is between 40 and 90. If the signal is excessively noisy increase lock power and reduce **lock gain** but keep in mind that lock power should *always* be *at least 10* below lock gain.

14. Maximize the lock level using the **lock phase** button in +/- 1 range.

15. Once the lock level is at a maximum, adjust **lock power** to maintain a lock level between 40 and 90.

16. Click Lock Scan to turn it off