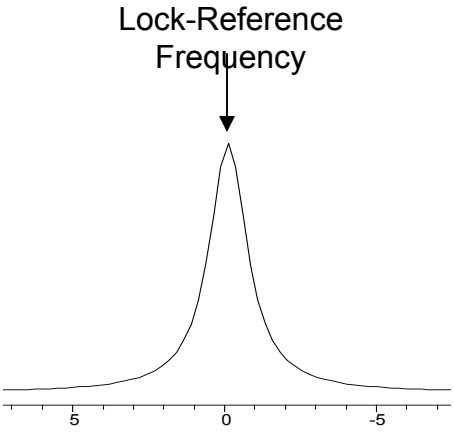
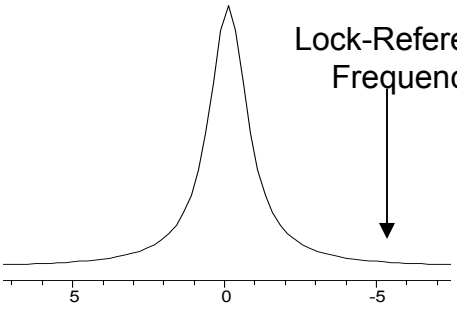
Locking and Shimming:

We use deuterated solvents to prevent unwanted solvent signals (^{1}H NMR); however, the deuterium (^{2}H) NMR signal is also used to ensure that the magnetic field doesn't change during the NMR experiment. The frequency of the ^{2}H NMR from the solvent deuterium essentially "*locks*" the magnetic field.

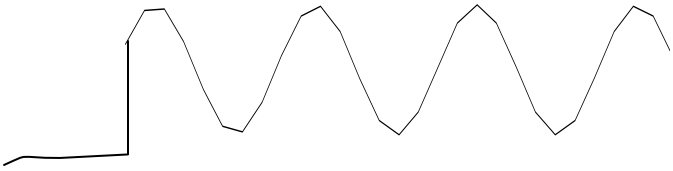
Once Locked, the lock-circuit assumes control over the field-offset (or *Z0* on a Varian instrument), and while locked the magnetic field cannot drift, even for long-term acquisitions.



Lock-Display on a Varian Instrument
(When Locked, *On-Resonance)*



Lock Display on a Varian Instrument when field is offset (Z0 needs to be changed).



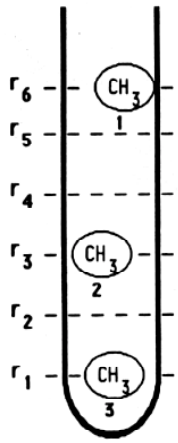
The Lock-Level corresponds to the *height* of the lock (^{2}H) NMR signal. Since the area of the lock signal is constant (for a particular lock-power/lock-gain setting), a narrow (sharp) lock peak will be taller (i.e. larger lock level), and a broad lock signal will be shorter (i.e. lower lock level). Therefore, the lock-level is an indicator of how broad or sharp the peak (of the ^{2}H solvent signal) is.

Note: If the lock-level drifts up and down, this is indicative of the Lock-Power being set too high. Excessive lock-power leads to saturation of the lock signal, and a slowly oscillating lock level.

Shimming the magnetic field:

To achieve good resolution, the magnetic field must be *homogeneous*; however, the sample distorts the magnetic field. The "*shims"* are special coils surrounding the sample probe, which allow one to re-shape the magnetic field. Good shimming (achieving an optimal, homogeneous magnetic field) is key to getting a good, high-resolution NMR spectrum.

Example: A Magnetic Field Gradient (field-strength varies vs. position in the NMR tube.

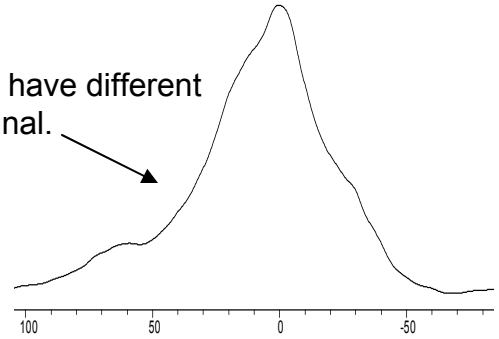


$B(r_6) > B(r_5) ... > B(r_1)$

Higher Field

$CH_3(1)$, $CH_3(2)$, and $CH_3(3)$ all have different frequencies, giving a broad signal.

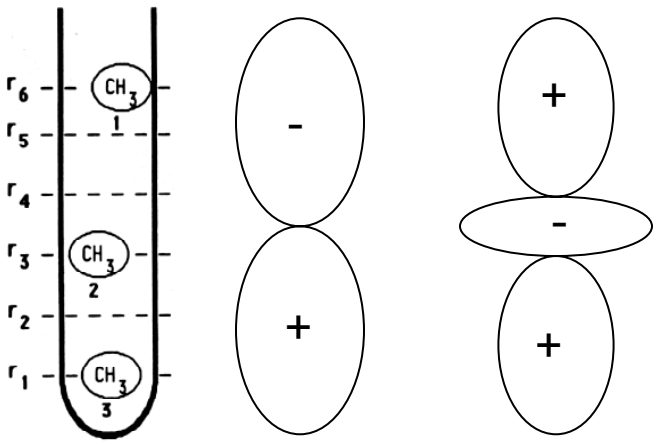
Lower Field

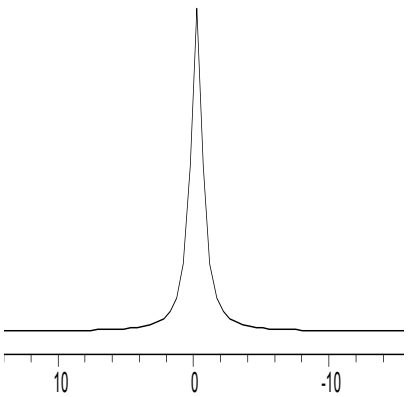


Properly adjusting the currents in the *shims* will "fill-in" the inhomogeneities, hopefully yielding a homogeneous field, and sharp, symmetric peak lineshapes.

Z1 Shim

Z2 Shim





Increasing lock-level is indicative of a narrower lock peak; therefore we adjust the shims to achieve the highest possible lock-level, which should correspond to the best possible lineshape.

Achieving optimal Lineshape by shimming Z1 and Z2:

Most people learn to shim by simply adjusting Z1 for maximum lock level, then adjusting Z2, and then back to Z1, ...etc. This process of simple iteration can (in fortunate cases) yield good results; however, this is not generally the best way to get truly optimal results.

If Z2 is badly mis-adjusted, one will never find the best values for Z1 and Z2 unless a "second-order" process is employed as follows:

- first, Z1 and Z2 are adjusted individually for maximum lock level.
- then, Z2 is deliberately moved in a known direction (either up or down), until the lock-level drops noticeably.
- Z1 is then re-optimized, and one must note if the new maximum is better (or worse) than the previous maximum. If the new maximum is better, then Z2 should be moved further in the same direction, until a global maximum is determined.
    - If this value for Z2 yields a lower maximum lock-level than previously observed the original value for Z2, this means that Z2 was moved the wrong way...move Z2 the opposite direction, re-optimize Z1, to ultimately find the global maximum in the lock level (as determined by Z1 and Z2).
- Sometimes, Z2 must be moved a large amount to find the best (global) maximum in the lock level.

When using short (low-volume) samples, such that the edges of the sample get close to the edges of the detection coil in the probe, Z2 requires major adjustment. For these samples, expect a very large change in Z2, and it is extremely critical that the sample be centered above/below the coil-center.

The Z3 shim requires a "double-second-order" process for proper adjustment. In other words, one must move Z3, and then go through the second-order process for optimizing Z2 described above. Because of this, only Auto-Shim should be used for Z3 or (preferably) gradient-shimming when available.

-R.Shoemaker