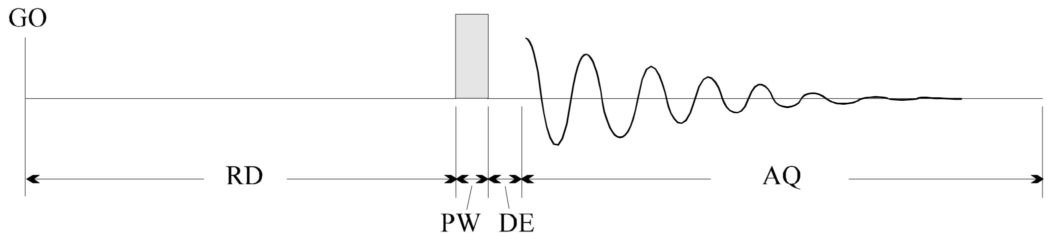
2. Bruker Acquisition Basics

by *cg fry:* created 2/17/94 – updated 12-Sep-00



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I. Description

[See the Primers chapter for more specific information on certain areas]

Acquisition on Bruker AM and AC spectrometers is based on the command **GO.** **GS** (go-setup), **ZG** (zero-go), and **CO** (continue) all use the **GO** command; **ZG** zeroes memory and coadds acquisitions until **NS** and **NE** are fullfilled, whereas **CO** coadds new acquisitions to the current memory. **GS** displays only the most recent acquisition.

Four events are initiated when **GO** is issued, as shown in the figure above:

i) The spectrometer waits a time <u>RD</u> seconds; relaxation delay.

ii) A strong transverse rf pulse is applied with a duration <u>PW</u> µs.

iii) A delay of duration <u>DE</u> µs occurs to allow for probe ringdown.

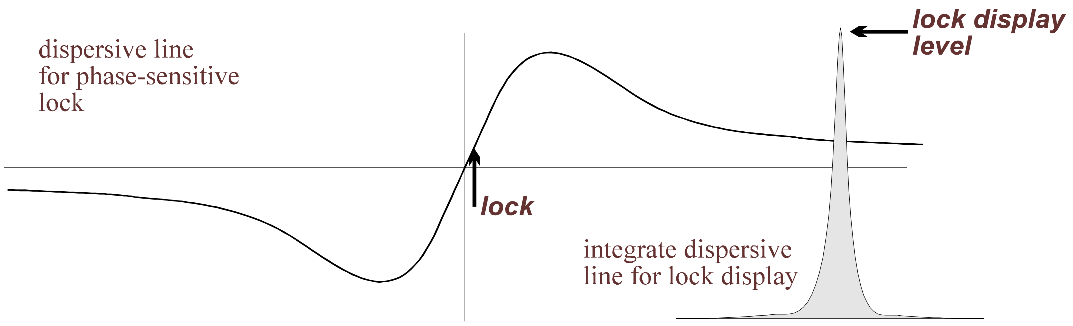
iv) Acquisition begins, and lasts a time <u>AQ</u> seconds.

II. Deuterium Lock

Locking on a deuterated solvent involves adjusting four spectrometer variables:

i) Change ***Z0 shim*** (**FIELD**) to find the lock signal. Deuterons track closely with protons, so $CDCl_3$ will be 5.2 ppm downfield of $(CD_3)_2CO$. It should be clear (if you don't know, please ask!) that changing the magnetic field strength by 5.2 ppm will require a change in the spectrometer frequency by the same amount to keep a 10 ppm sweep width running from TMS at δ=0 ppm to δ=10 ppm.

ii) The second adjustment involves the ***lock phase.*** The lock uses a *phase-sensitive* loop to keep the frequency of the spectrometer stable:



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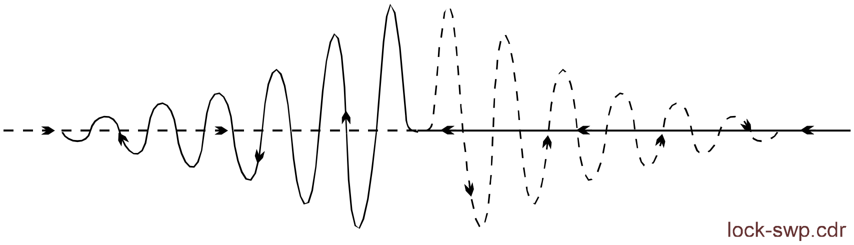
The lock in sweep mode on Bruker spectrometers should always have a positive initial slope.

Correct phase on lock sweep display
(use Dual Display on SCM unit)



Changes in solvent, temperature, and even in adjustments from poor to good shims can change the phase. *Good lock phase is critical to obtaining a stable, good lock, and thus a good shim and lineshape!* So you should adjust the lock phase after any such changes. Initially use the sweep display, but later carefully adjust the lock phase to maximize the lock amplitude.

iii) The other two adjustments are the ***lock gain*** (lock *amp* on WP's) and ***lock power.*** Although the apparent effect of both controls is similar, to increase/decrease the lock intensity, the actual changes are fundamentally different. The ***lock gain*** is the lock receiver gain, changing nothing involved with the sample magnetization. The ***lock gain*** can therefore be changed at will with little danger.

iv) The ***lock power,*** on the other hand, must be carefully adjusted to achieve the best lock performance. This control changes the radiofrequency (rf) power to the sample. Too little power will give a weak response, and a noisy lock. Too much power will cause saturation, which *must* be avoided. Proper settings can be found for any solvent using the following technique:

> Increase/decrease the ***lock power,*** and watch for a "bounce" in the signal: the signal will go up/down in response to the change in power, but if it then rebounds (perhaps just slightly) down/up as in a bounce, the power is too high. Decrease the power until the bounce cannot be observed, and decrease the power another 20% to be safe.

Suggested settings for the ***lock power*** for the most common solvents are posted next to each AC spectrometer in the facility.

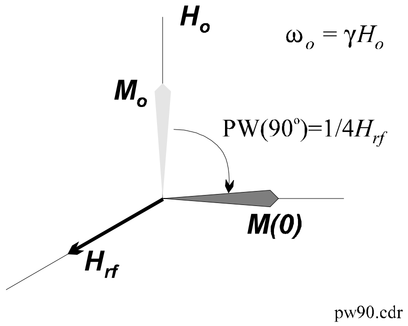
III. Shimming

Once lock is achieved, the magnetic field inhomogeniety must be shimmed out. For experimental planning, one must keep in mind differences in solvents. Acetone-d_6 has a very narrow natural ^2H linewidth, and therefore gives good performance for shimming. DMSO-d_6 and pyridine-d_7 both have much broader natural ^2H linewidths, and are therefore relatively poor solvents for lock shimming. D_2O can give very broad ^2H lines due to exchange. Of course, the last three solvents might be the only choices for solvation and cost reasons, but shimming on the FID might then be necessary if high-resolution ^1H spectra are needed.

A number of good discussions about shimming strategies are available. Since every change of sample requires shimming, every spectroscopist should take advantage of strategies to lessen the time spent shimming. Especially useful is G. A. Pearson, "Shimming an NMR Magnet," Chem. Dept., Univ. IA, Iowa City, IA 52252. Many practical examples are given that show how to visually determine which shim needs adjusting, and in which direction. These quick determinations can save much time at the spectrometer and lead to improved spectra. More detailed discussions of shimming can be found in Derome, p. 42-50, and in G. N. Chmurny and D. I. Hoult, "The Ancient and Honorable Art of Shimming," Concepts Magn. Reson. **2,** 131-149 (1990).

IV. Acquisition Parameters

The most important acquisition parameters are briefly described in this section. The mnemonics are specific to Bruker spectrometers:



PW – pulse width of <u>GO</u> command (see Section I)

RD – relaxation delay

DE – probe/filter ringdown delay

SW – sweep width in Hz

O1 – sets center of spectrum by changing absolute frequency

DW – dwell time in µs; time per digitized point; set by SW → $DW = 1/2SW$

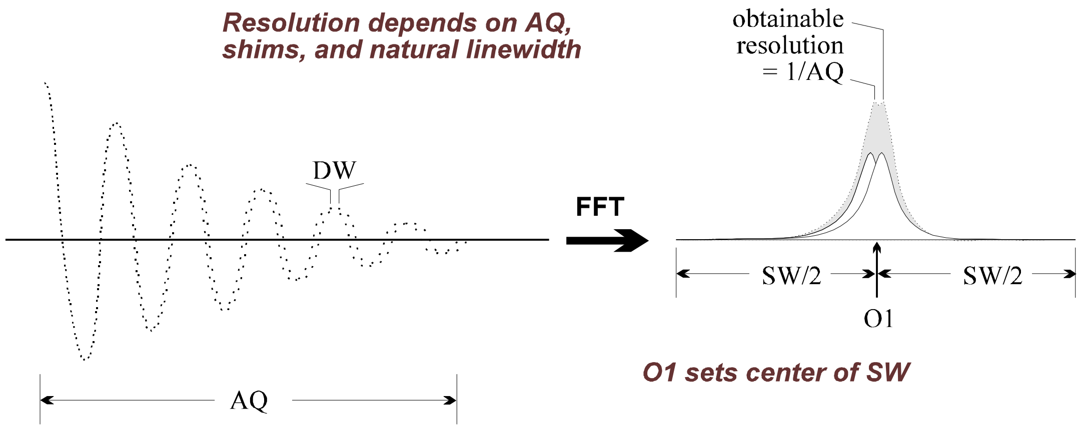
TD – data acquisition size; number of points digitized; TD/2 = # complex data pairs

SI – Fourier transform size; zero fills if TD<SI; only half SI used for real part, so

$$\text{\# points in spectrum} = SI/2 \qquad \text{digital resolution} = \frac{\text{sweep width}}{\text{\# points in spectrum}} = \frac{SW}{SI/2} = \frac{TD}{AQ \cdot SI}$$

AQ – acquisition time; related to *obtainable resolution* (i.e., best resolution possible) unless sophisticated analytical procedures, such as linear prediction, are used. The digital resolution can be improved by zero-filling, but not the actual obtainable resolution (appropriate Gaussian multiplication perhaps can take advantage of one zero-fill, but further zero-fills will not help).

$$\text{obtainable resolution} = \frac{1}{AQ} \qquad AQ = DW \cdot TD = \frac{TD}{2SW}$$



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