II. ¹*H* pw90 Calibration

[updated: 29 June 2010]

- 1. Acquire a normal ${}^{1}H$ 1d spectrum, as described in Section I (sbs_1H1d).
- 2. Check the value of **pw90** brought in from the SETUP macro/button with: **pw90?**
- 3. Until you are confident about this calibration, start with a coarse array as:

array₊J

parameter to be arrayed: \mathbf{pw}_{+} enter number of steps in array: $\mathbf{30}_{+}$ enter starting value: $\mathbf{3}_{+}$ enter array increment: $\mathbf{3}_{+}$

- the steps above can be entered in a single command: array('pw',30,3,3),
- 4. Make the following changes: $nt=1 ss=0 vp=80 at=2 fn=32k d1=1 \downarrow$
- 4. go, J dsa, will start the acquisition and update the display after each fid is acquired (on the UNITY, wait a few sec after go, before issuing the dsa,).
 - if the magnetization is not fairly close to sinusoidal (is slow to invert), **d1** needs to be increased (i.e., the ${}^{I}HT_{I}$ relaxation is slow).
- 5. Determine the 360° crossover position:
 - da_{\rightarrow} and dg_{\rightarrow} display the array and main parameter group, respectively.
 - wft dssh dssl→ will retransform, display spectra stacked horizontally, and label the spectra, respectively.
 - **ff** \downarrow (= **f full**) will expand one spectrum to show the full spectrum using the full screen (respectively).
 - wft(3) ds(3) df(3) will transform, display spectrum and display fid of the 3rd "FID" (array element), respectively.
 - **pw[3]?** will display the 3rd element of the pw array.
 - pl('all')→ and pl(1,100,10)→ will plot the full array, or the 1st, 11th, 21st, ... to 91st spectrum using current stack plot parameters (do a dssa or dssh 1st)
- 6. Perform a fine array about the 360° position:
 - as an example, suppose the 360° crossover observed in section 4 is ~47, then
 array('pw',20,44,0.5), gives a good fine array.
 - again, use $go \downarrow dsa \downarrow$ to acquire and display the data.
- 7. Determine the best crossover position, then supposing it gives 48.5:

pw90 = 48.5/4 pw=pw90 go.J

- the last **go** checks that **gain** is not set too high.