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Pulse Width (pw90) Determination

You can determine the pulse width (pw) on any spectrometer using Vnmr software by using the procedure described here. Typically, the 360-degree pulse width (pw360) is determined, and divided by 4 to compute the pw90, which will yield maximum S/N if used for the pw value. ***The value for pw90 is instrument-dependent.***

Set-Up

This particular example pertains to values typical on the UI500NB (pw90 is usually about 6.5 μsec).

ga Acquires a spectrum under default conditions. Usually nt=1 is sufficient, but you can increase it.
aph Autophase or phase properly if needed.
gain? Note the gain. Then, type **gain =** (original gain value – 6 units).

vp = 70 Places spectrum about half-way up on the display window.
vsadj Adjusts the vertical scale so that an acquisition made with pw ≈ pw90 is scaled properly.
ai Sets **a**bsolute **i**ntensity mode so peaks do not individually rescale.

You should perform this determination using an NMR tube containing *your sample and solvent*. Narrow the viewing window to a peak in about the middle of your spectrum. The strategy in *using a mid-range peak* is to select a peak to measure which is typical of the distribution of peaks in your analyte molecule. Don't use the solvent peak – it usually relaxes too slowly, and it is not your analyte.

d1 = 10 Sets a delay of 10 sec between acquisitions in the array to allow for relaxation between acquisitions. Helps make the array sinusoidal, which it should be if d1 is large enough.

Now, type **array**

The next four items are the answers to the questions posed by the **array** macro. The values here are reasonable for demonstration purposes and cover the range from 0° to past 360°. The PW90 is usually about 8.9 μs.

- Parameter to be arrayed: **pw**
- Enter number of steps in array (decide on something reasonable): **17**
- Starting value (you decide): **0** (value is in μsec)
- Array increment (you decide): **2** (value is in μsec)

da **D**isplay **a**rray; displays current array values so you can review them.
dg **D**isplay **g**roup; displays the parameter group in the box at the bottom of the screen
ga Do not re-phase during data acquisition, tempting though it may be.

Analysis

dssh dssl **D**isplay **s**tacked **s**pectra **h**orizontally; **D**isplay **s**tacked **s**pectra **l**abels.

- As the spectra accumulate, they can be viewed with this pair of commands.
- Use **ds(#)** to display a desired spectrum by its increment number, such as ds(4)
- If you have an understanding of where the pw360 is, you can start your array just before that pw value, and proceed through the null.
- You can terminate the experiment with **aa** once you have unambiguously determined the pw360.
- **pw90** = (the numeric value for pw360)/4 [that is, pw90=29/4 will compute pw90 for you by dividing]
- **pw = pw90**
    - Sets pw to pw90 if desired; typically done on the UI500NB as default
- To print the array, type **pl(1,17) pap page** and you should get a non-distorted sine wave. If not, add 5 sec to d1, and re-generate the array by typing **ga** again and re-collect the data.
- You can save the entire array of spectra if desired. Just type **svf** and save as usual.
    - A good name is **pw90cal.quinine.cdcl3**
- To fine-tune conditions for pw360 determination, you can run the array in a range more tightly defined about the 360 degree null.

Finish

- Once complete, please re-tune and re-match on $CDCl_3$ if you used a different solvent.
- In other words, leave the UI500NB tuned and matched to $CDCl_3$ for future users.