

Determination of the 90° Pulse Width

The 90° pulse-width ($pw90$) is the length of a pulse, usually in microseconds, that will produce the maximum response from a given NMR-active nucleus. The value of $pw90$ depends on the nucleus, pulse power ($tpwr$), probe tuning, and the research sample. Routine acquisitions do not generally require calibration of $pw90$ because the default value determined by the NMR facility is sufficient to get reliable results. However, many advanced 1D and most 2D experiments rely on an accurately determined 90° pulse and thus, calibration is recommended. Luckily, calibration of the 90° pulse is straightforward. Since it is easier to determine a null point, we usually look for the 360° pulse, which gives a null spectrum (see Figure 1), and then divide the result by 4.

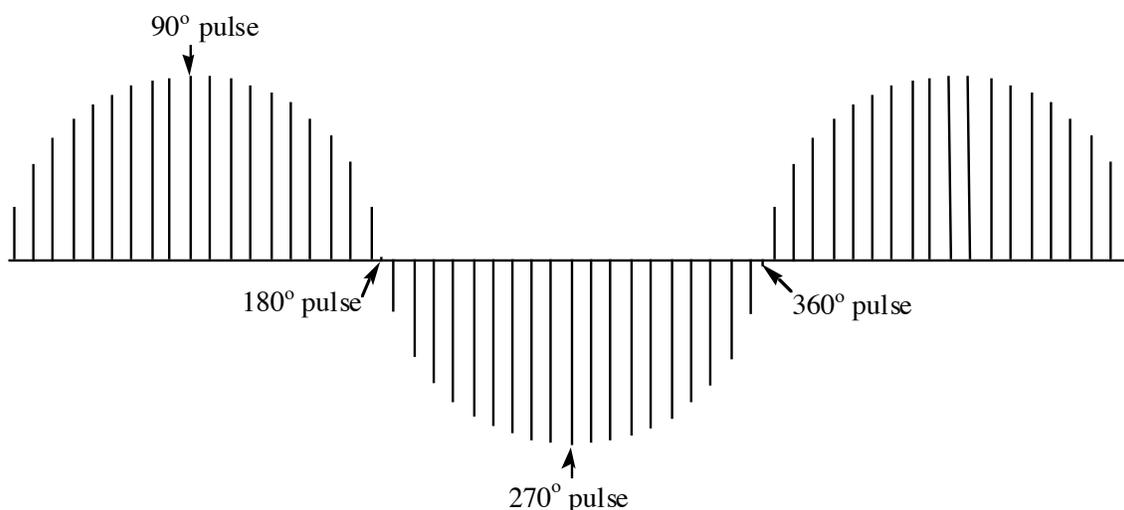


Figure 1. Representation of typical view of arrayed pw data for a single resonance. The null points are at 180° and 360°. Notice that determining the 90° pulse-width directly is more difficult than finding the null points.

Explanation of Types of Commands Found in this Handout:

1. The VNMR software and the UNIX operating system are both case sensitive. This means that the computer distinguishes whether the letters are entered in upper case (*i.e.* CAPITALS) or lower case. The user must be careful to type the correct case for each letter in a command.

Example: **jexp1** is not the same as **JEXP1**

2. Some commands are line commands and are typed in by the user followed by a return (a Return is assumed for typed **bold** text commands).

Example: **su**

3. Some commands are executed by clicking a mouse button with its pointer on a "button" found on the screen. The execution of these commands are indicated by a two letter designation (LC {left click}, RC {right click}, or CC {center click}) followed by a word or words in shadow or **bold** that would appear in the button.

Example: LC **Main Menu**

This means to click the left mouse button with its pointer on the button that says "Main Menu".

4. Some commands are executed by the mouse itself. These commands are indicated by a two letter designation (LC, RC, or CC) and a description of what the user should do in parentheses.

Example: LC (at 6 ppm)

This means to click the left mouse button with the mouse cursor positioned at 6ppm.

5. Parameters are entered by typing the parameter name followed by a equal sign, the value, and a return.

Example: **nt=16**

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TUNE THE PROBE

This applies to the probes on the VXR-500, Unity-plus 500, VXR-300, Inova-300, and Inova-600.

Refer to the guidelines posted on the magnet for probe tuning.

IMPORTANT: DO NOT TUNE THE AUTOSWITCHABLE QUAD PROBE ON THE INOVA-300 (RA IN THE SUBBASEMENT).

ACQUIRE A PRELIMINARY SPECTRUM

Setup a standard 1D experiment (e.g. H1, CDCl3).

Acquire, Fourier transform, and phase the resulting data.

SETUP AN ARRAY OF PW VALUES

pw90?

reports current value of the 90° pulse-width. Please note this value.
setup an arrayed experiment.

array

You will need to answer the following questions, which will appear above the command line:

parameter to be arrayed:
pw
enter number of steps in array:
10
enter starting value:
enter a value = (4*pw90)-1 from above

pulse width. You want to find the 360° pulse width, which gives null peak heights.

this is the number of different points you will check for the 360° pulse.

pw90 is from the command *pw90?*. A value around 31 would be average. NOTE that this starting value is dependent upon the salt concentration of the sample. If it is hard to determine the *pw90*, try arraying the *pw* in 20 steps with a starting value of 5 and increment by 5.

enter array increment:
0.5

this is the step increase between each point

pw[1]=1

this will replace the first array element with 1. A 1 μs pulse will be positive and will aid in phasing the arrayed data.

da

displays the array

RUN 90° PULSE-WIDTH DETERMINATION
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ds display spectrum

Expand around any well-resolved resonance.

gain='y' turns automatic gain control off, which is not allowed for arrayed experiments.

d1=3 set recycle delay to 3 seconds. *d1 + at* should be about 1 to 1.5 times your longest T_1 .

<p>NOTE: If your T_1's are long, you can try the setting of $d1=3$. If you set $d1$ for less than the recommended time, you will sacrifice sensitivity. However, if your sample concentration is adequate, the savings in time will more than balance this loss.</p>
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go start the arrayed experiment.
ai absolute intensity (i.e. no peaks heights are scaled).
wft Fourier transform the FID.
dssh display stacked plots horizontally.

The first spectrum will be positive and the second should be negative. If this is not the case, rerun the array starting with a lower starting value (e.g. 28).

dssl Look for the spectrum that has the lowest peak intensities. Note the value: _____
 for example, **pw90=33.5/4**.
pw90=your determined value/4 reset pulse width to equal the 90° pulse.
pw=pw90 Returns the pulse width.
pw? turns autogain on.
gain='n' acquire a spectrum with new *pw*.
ga

Now you can setup your advanced experiment using this new pulse width.