Calibration of 90° Pulse Lengths

PART I – INTRODUCTION

1. Occasionally, some experiments will require more careful pulse length calibration.
2. This handout will teach you how to calibrate the 90° pulse for ^{1}H (or other nuclei). It assumes basic familiarity with running a routine 1D NMR experiment.

Do all of the usual set-up stuff

1) You will need to do the usual things: log on, insert your sample, create a new experiment, read in the parameters, lock, shim, tune, etc.

PART II – CALIBRATE ^{1}H 90° PULSE

1. Automatic 90° pulse Calibration

new

1) Type **new** to set up a new experiment. Select the proton experiment from the panel on the right side of Topspin or type **rpar** to read in the proton parameters.

rpar

pulsecal

2) Execute the automatic 90° pulse calculation by running the command **pulsecal**. Pulsecal returns three values. Take note of the bottom one, your 90° pulse (μs) and the power level (dB) (Fig 1.) If pulsecal fails ("too many entries in peaklist"), proceed to manual calibration.

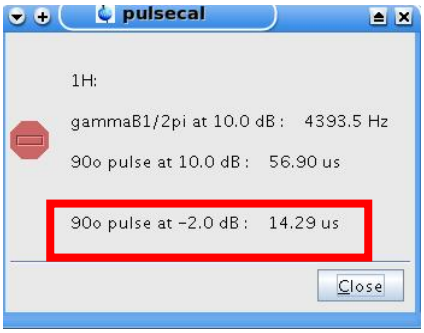

Figure 1: Pulsecal output. Use the bottom value as your 90° pulse.

2. Quick Manual 90° Pulse Calibration

pulprog zg

1) Follow steps 1 and 2 of the Automatic Pulse Calibration procedure above.
2) Set the pulse program **pulprog** to zg (should have been set as zg30 before).

getprosol

3) Type **getprosol** to ensure that the most recently determined values for pulse times and power levels for all nuclei in the particular pulse program will be used.

rga

3) Type **rga** to start an automatic receiver gain set routine. When this is finished, the message **rga:finished** is output above the pink command line. Record the number that is set as **rg**.

ns 1
ds 0
zg
ef
apk

4) Set **ns 1**, **ds 0**.
5) Execute the experiment by running the command **zg**. After the experiment is done, process the data by running the commands **ef** (exponential multiplication and fourier transform) and **apk** (automatic phase correction).

change p1 to p1*4

6) Change **p1** to a value that is four times the initial value, e.g., from p1 = 10,

change to p1 = 40. You can do the math in the box by entering *4 after the current value. Rerun the experiment with **zg**.

a. After the experiment is done, process the data with e**fp.** Do NOT run apk! If you do, you will need to restart from step 3!.

b. Inspect the spectrum, then change **p1** and acquire again until you get approximately a null signal (equal parts positive and negative).

c. Make sure you wait at least 5 seconds between each acquisition to allow for sufficient relaxation.

d. The null signal is your 360° pulse. Divide the **p1** value corresponding to your null signal by 4 to get the 90° pulse. To save keystrokes, you can use the command **zefp**, which combines **zg** + **efp** and **overwrites your data without asking**.

7) Make note of the **p1** value for your 2D or other experiments.

or you can use paropt

3. 90° Pulse Calibration with paropt

1) Alternatively, you can use the *paropt* command to calibrate your 90° pulse length. This will give you a full sinusoidal pulse calibration profile.

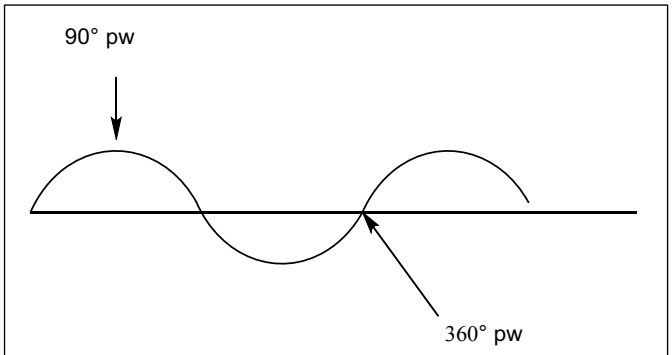

Figure 2: Sinusoidal Pulse Calibration Profile

2) Follow steps 1-4 of the Quick Manual Calibration step above

set p1 = p1/2

3) Set p1 to p1/2 (e.g., if p1 = 14 after getprosol, set p1 = 7).

zg; ef; apk

4) Acquire a spectrum with **zg**, then **ef; apk**.

5) Zoom in on the peak you with to calculate the 90° pulse for.

dpl1

6) Type **dpl1**. This will set the left and right x-coordinates (F1/F2 or F1P/F2P) and the vertical scaling of the result.

paropt and follow the prompts

7) Type **paropt** and enter p1, the first value, the increment, and the number of measurements in the series. The first scan should be set to a few microseconds less than p1*4. Increments of a few microseconds is usually good enough.

8) Note: Each time paropt runs, you are left in procno 999. You can return to the expno and procno from which you started by using the re command (i.e., if you started in expno 1 procno 1, type re 1 1).

Look for the null point. That is your 360° pulse; the 90° is the p1 value corresponding to the null point divided by 4.

9) Expand the vertical scale as necessary to judge the null point as the point with symmetrically distributed residual signal. You can either use the

kill if you need to

scaling buttons on the left or the equivalent command line commands to rescale the display while paropt is running. You'll have to mentally count the number of increments over to the target peak, so choose start values and increments that make the arithmetic easy.

a. If you do not want to wait for the full array to run, you can **kill** the experiment. Type **kill**, and a window of all of the running processes will pop-up. Select *paropt* and hit the "Kill" button. Exit out of the pop-up window.

9) Consult one of the NMR staff or the Bruker manual if you have any further questions on paropt.

PART III – CALIBRATE A 90^{O} PULSE FOR ANOTHER NUCLEUS

1) You can use any of the above methods to calibrate pulse lengths for other nuclei.
2) However, if you do not have enough SNR to do a single scan, you must use labeled compounds for low abundance nuclei and signal average. This means you will need to change your d1 value appropriately to account for the T_1 relaxation of your compound! Contact facility staff to setup pulse parameters for currently unused nuclei.