# Determination of the Proton 90° Pulse
Revised: 4-17-2021

The 90° pulse can be determined by either looking for the 180° pulse or the 360° pulse – both give a null signal. Using the 360° pulse results in a more accurate measurement. Here we will run an approximate determination looking for a 180° pulse, and then we will fine tune the determination by measuring a 360° pulse.

1. Obtain a normal one scan proton spectrum of your sample. Select the *AcquPars* window and set **NS**=1 and **DS**=0. If you have previously acquired a proton spectrum of your sample, you can load that spectrum, instead.

2. Identify a peak near the center of the spectrum to focus on in the determination. Singlets are nice to use, but any strong peak will do. Expand around that peak and set the cursor in the middle of the peak. Leave a 1-2 tenths of a ppm on either side of the peak in your expansion.

3. Click the *O1 Adjust* button: The dialog box to the right pops up.
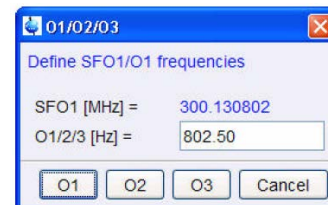

Place the cursor on the peak, and then in the dialog box, click on **O1** to set the O1 to the cursor position. This places your peak in the exact center of the spectral window (SW).

4. Right click on the spectrum and select *Save Display Region To…* in the drop-down menu.

   Doing so opens the dialog box shown. Select the *Parameters F1/2* option and click **OK**. The purpose of this step is to define the spectral region that will be displayed in the plot of the spectra acquired in the experiment.
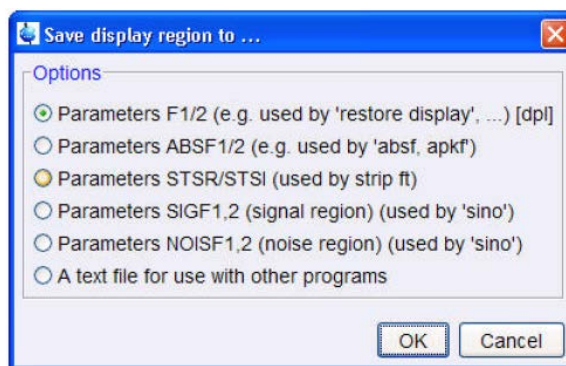
5. Select the *AcquPars* window and change the following parameters:
   - **PULPROG = zg** (uses P1 directly, nominally the 90° pulse, rather than zg30 which is a 30° pulse – P1*0.33).
   - **D1** = 25 (ideally, this relaxation delay would be set to 5*T1 of the longest relaxing proton to allow complete relaxation between the experiments. Since it is not unheard of for protons to have 5-6 second T1s, set D1 longer if you have the time to spare.
   - **DS** = 0
   - **NS** = 1

6. Select the *ProcPars* window and change **PH_mod** to "**pk**". This sets up automatic phasing of the spectra, applying the same phase correction to all spectra (so that all are not automatically adjusted with positive phasing).

7. Type *popt* to open the table where you will input the experimental parameters for the arrayed parameter (P1). The dialog box shown below opens.
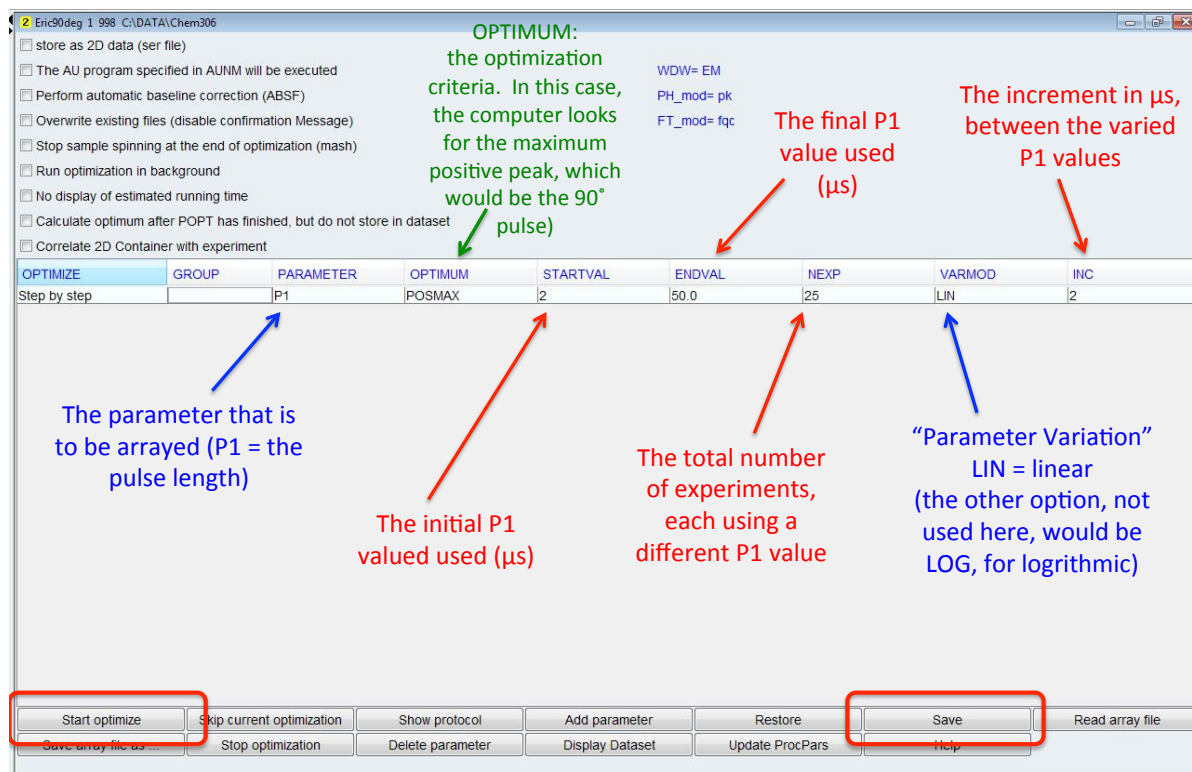

Input the following values into the table:

- **PARAMETER** = P1 (P1 is the arrayed parameter)
- **OPTIMUM** = POSMAX
- **STARTVAL** = 2 (the first experiment will use P1 = 2 μs)
- **ENDVAL** = 50 (the last experiment will use P1 = 50 μs)
- **VARMOD** should be **LIN** (linear as opposed to logarithmic variation in the P1 value)
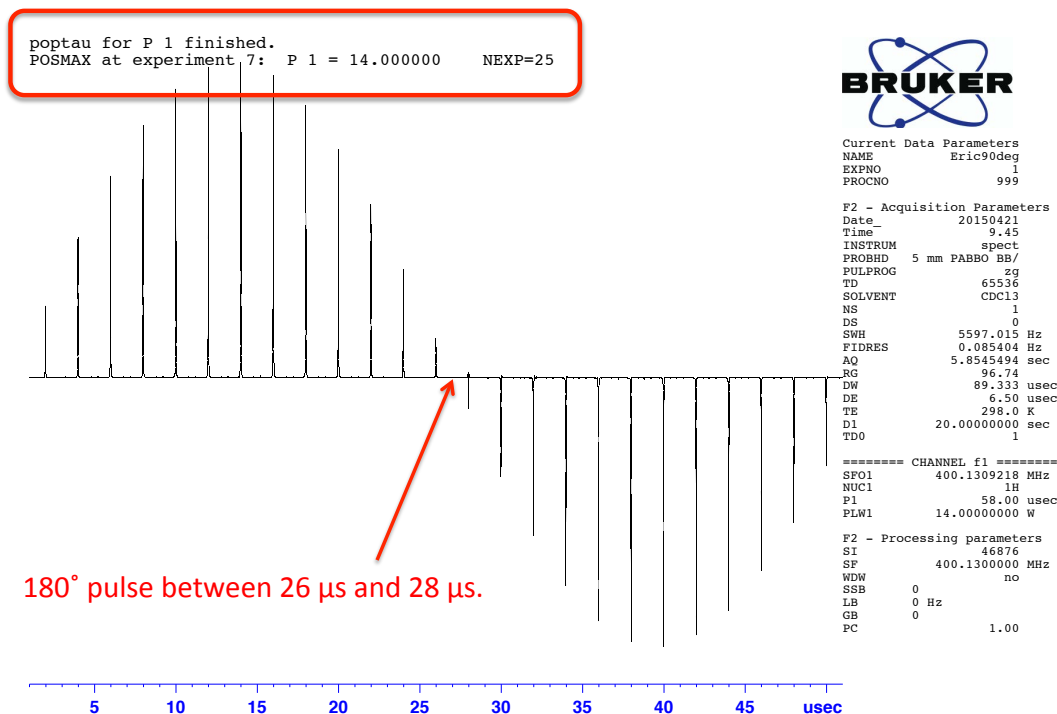- **INC** = 2 (the increment between P1 values)

Note: Setting INC will determine (and set) the value in the NEXP cell (the number of experiments). Thus, STARTVAL, ENDVAL, NEXP and INC are all dependent on one another.

Thus, with these values, the experiment will involve 25 different FIDs, with P1 varied through the resulting spectra from 2 μs to 50 μs, incremented by 2 μs each time.

8. Make sure all cells are populated (except for **GROUP**, Which the computer will take care of). Click on **Save** and then **Start Optimize**, to initiate the experiment.

9. The *poptau* dialog box will appear (shown to the right). Enter "y" to continue.

10. When the experiment is complete, the nominal 90° pulse will be displayed (the computer simply choose the value of P1 that gave the maximum peak. Depending on the parameters you chose in step 7, this may be far off from
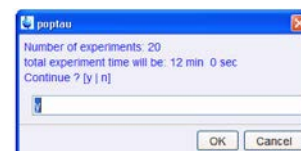
the correct value. Select the *Spectrum* window to see the entire array of spectra, as shown below. Click the spectrum re-center button to center and scale the plot. The computer's 90° pulse determination is shown at the top of the arrayed plot. Note that the EXPNO for this experiment is 999.

```
poptau for P 1 finished.
POSMAX at experiment 7:   P 1 = 14.000000      NEXP=25
```

BRUKER

```
Current Data Parameters
NAME              Eric90deg
EXPNO                     1
PROCNO                  999

F2 - Acquisition Parameters
Date_              20150421
Time                   9.45
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg
TD                    65536
SOLVENT               CDCl3
NS                        1
DS                        0
SWH              5597.015 Hz
FIDRES           0.085404 Hz
AQ             5.8545494 sec
RG                    96.74
DW                89.333 usec
DE                 6.50 usec
TE                  298.0 K
D1          20.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1        400.1309218 MHz
NUC1                     1H
P1                58.00 usec
PLW1         14.00000000 W

F2 - Processing parameters
SI                    46876
SF          400.1300000 MHz
WDW                      no
SSB          0
LB           0 Hz
GB           0
PC                     1.00
```

180˚ pulse between 26 μs and 28 μs.

5   10   15   20   25   30   35   40   45   **usec**

To estimate the 90° pulse, look for the P1 value that gives a 180° pulse – the cross over point where the spectrum goes from positive to negative peaks. You can see in the example that the 180° pulse is about 27.5 μs.

### *Fine tuning the determination – looking for a 360˚ pulse.*

If the 180° pulse is about 27.5 μs, the 360° pulse is about 55. To fine tune the calibration, set up a 360° pulse determination, but this time in steps of 0.5 μs.

1. Type *popt* to open the arrayed parameter table again. Here are reasonable values to use for *this example*, with a ~55 μs 360° pulse. **In your case, choose STARTVAL and ENDVAL that straddle your approximate 360˚ pulse value.**

    -   **STARTVAL** = 50 (the first experiment will use P1 = 50 μs)
    -   **ENDVAL** = 60 (the last experiment will use P1 = 60 μs)
    -   **INC** = 0.5 (the increment between P1 values)

    NEXP should change to 20, based on these values. Might be a slightly different value depending on your values.

2. Click on **Save** and then **Start Optimize**, to initiate the experiment.

3. The *poptau* dialog box will again appear, as will a series of dialog boxes. Most ask about overwriting or appending files. Chose to append instead of overwriting the files from the 180° pulse determination. The combined spectra from the 180° pulse determination were stored in directory "999". Chose not to overwrite this file, you'll be asked if you want to create a new directory for the 360° pulse experiment in "998". Answer "y" to this. The file structure for the two pulse determination experiments is shown below.

4. The plot for this fine-tuned experiment will appear as below. The crossover point is the 360° pulse. Divide by 4 to get the value of the 90° pulse.

5. Click on the Plot button, found among the buttons running across the window above this spectrum. In the window that opens, you'll have to change the printer paper size from "A4" to regular "Letter" size, then print.
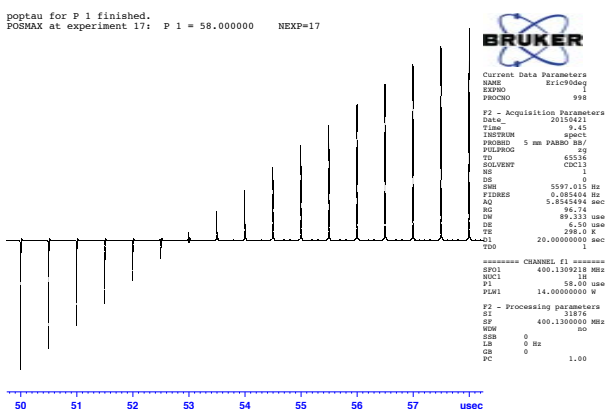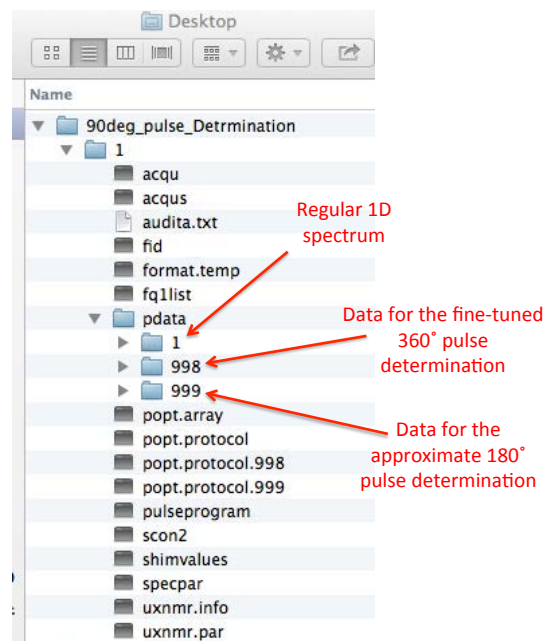
Optional: Processing the data in MNova

To open the files in MNova, drag your data directory to the local computer. In the finder, open the directory and you'll see a folder, probably called *"1"* (see the figure above). Open that folder and you'll see a number of files, including a folder called *"pdata"*. Open *pdata* and you'll see three directories (if you did both the 180 and 360 determinations). Those directories are named *1*, *998*, and *999*. Directory *1* contains the normal 1D spectrum of the sample, with the SW and O1 parameters you used. Directory *999* contains the approximate 180° pulse experiment data. Directory *998* contains the data for the find-tuned 360° pulse data. Drag the folders 998 and 999 onto MNova and you'll see your spectra plotted as shown something like the example below (in the example, I've stacked the results from the two experiments).