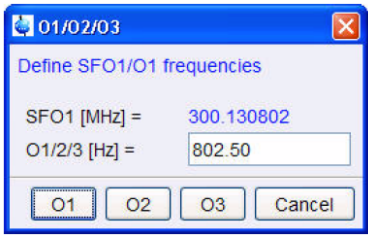
Determination of the Proton 90˚ Pulse

Revised: 4-11-2016

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.

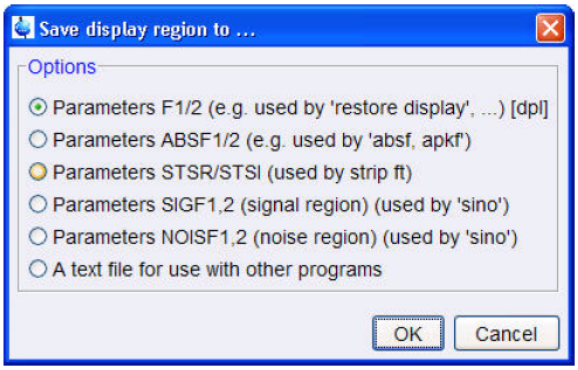
1. Obtain a normal one scan proton spectrum of your sample. Select the *AcquPars* window and set **NS**=1 and **DS**=0. If you have previously acquired a proton spectrum of your sample, you can load that spectrum, instead.
2. Identify a peak near the center of the spectrum to focus on in the determination. 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. With the cursor placed on the center of the peak, click the *O1 Adjust* button.



In the dialog box that pops up, 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.

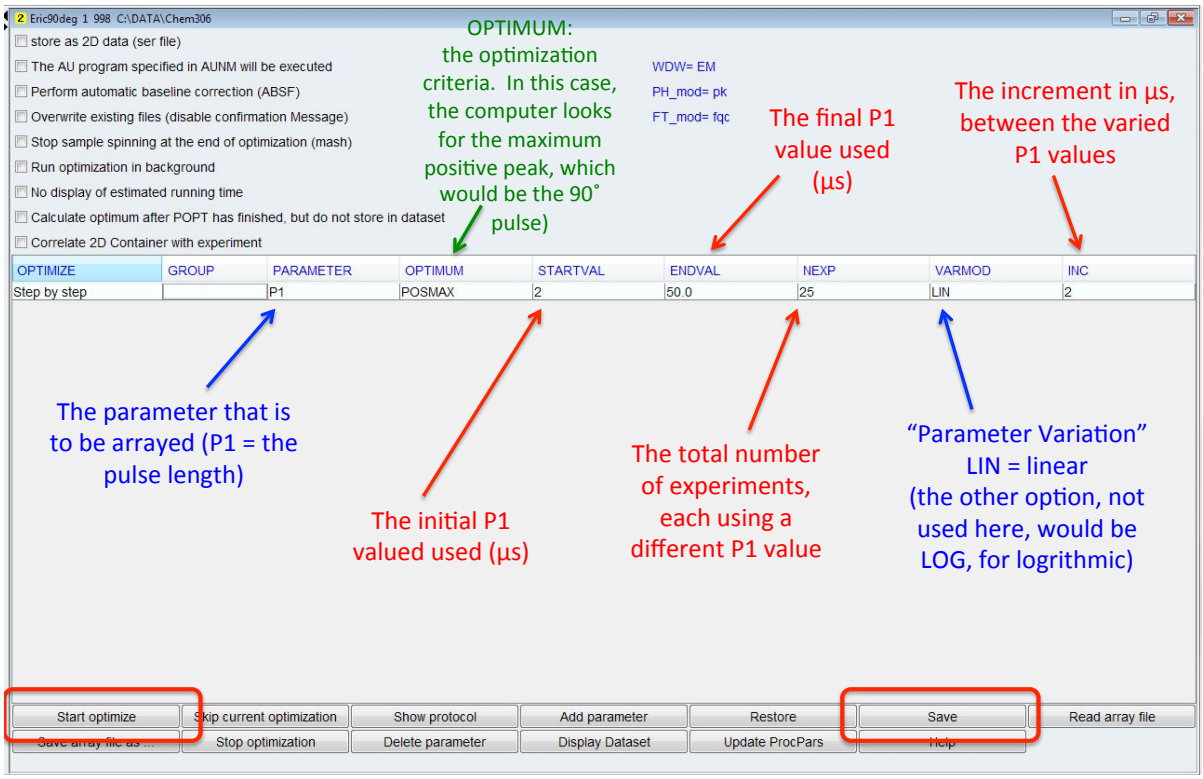
   Doing so opens the dialog box shown. Select the *Parameters F1/2* option and click **OK**. The purpose of this step is to define the spectral region that will be displayed in the plot of the spectra acquired in the experiment.



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:

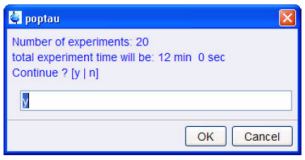
- **PARAMETER** = P1 (P1 is the arrayed parameter)
- **OPTIMUM** = POSMAX
- **STARTVAL** = 2 (the first experiment will use P1 = 2 μs)
- **ENDVAL** = 50 (the last experiment will use P1 = 50 μs)
- **VARMOD** should be **LIN** (linear as opposed to logarithmic variation in the P1 value)
- **INC** = 2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

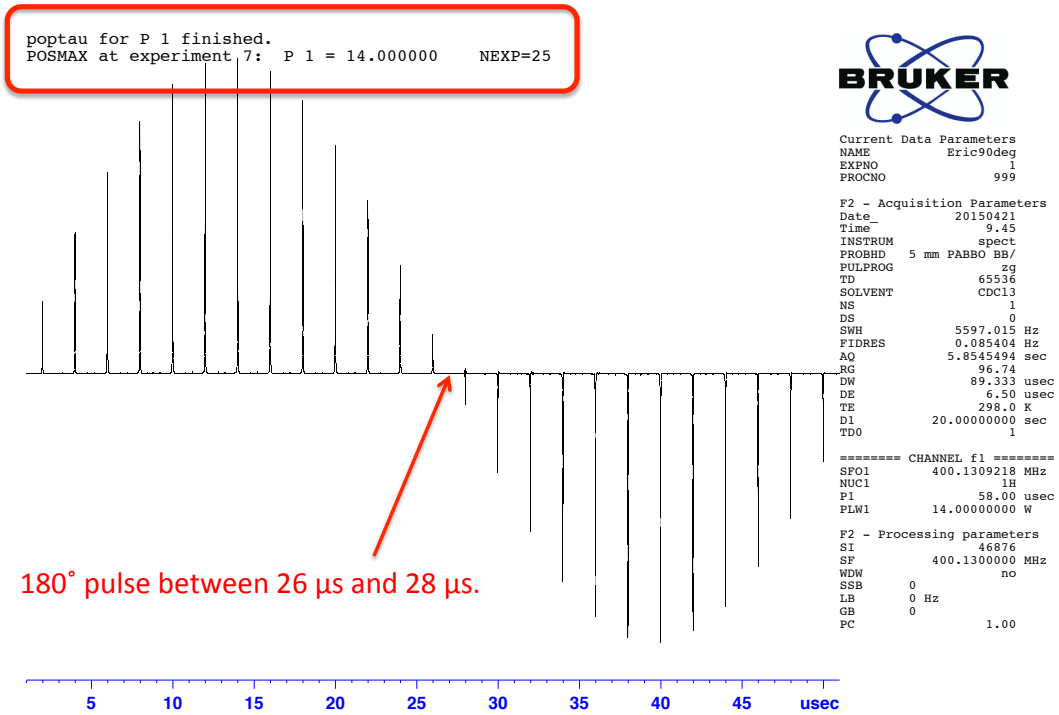
8. Make sure all cells are populated (except for **GROUP**, Which the computer will take care of). Click on **Save** and then **Start Optimize**, to initiate the experiment.

9. The *poptau* dialog box will appear. Enter "y" to overwrite old data, then click **OK**.



10. When the experiment is 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 or may not be close to the correct value. Select the *Spectrum* window to see the entire array of spectra, as shown

below. Click the spectrum reset 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.



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, 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. Change the following values:
   - **STARTVAL** = 50 (the first experiment will use P1 = 2 μs)
   - **ENDVAL** = 60 (the last experiment will use P1 = 50 μs)
   - **INC** = 0.5 (the increment between P1 values)

   NEXP should change to 20, based on these values.
2. Click on **Save** and then **Start Optimize**, to initiate the experiment.
3. The *poptau* dialog box will again appear. Enter "y" to overwrite old data, then click **OK**. Proceed as above. The plot for this fine-tuned experiment will appear as below.

Note: once more more dialog boxes may appear asking if you want to overwrite data (I can't recall them, as I write this). Answer with what seems appropriate. One will tell you, I think, that the data for this will be stored in EXPNO 998, which is appropriate….. Sorry, I'll fix this step soon!

4. The crossover point is the 360˚ pulse. Divide by 4 to get the value of the 90˚ pulse.