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Vanderbilt NMR Facilities

Instructions for 90° Pulse Width Determination

Using TOPSPIN

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1. In General

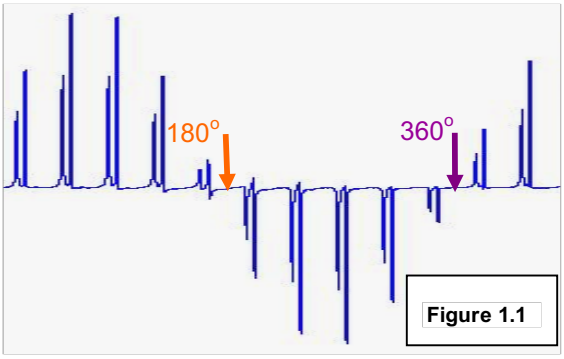


Figure 1.1

The NMR excitation profile is a sine wave

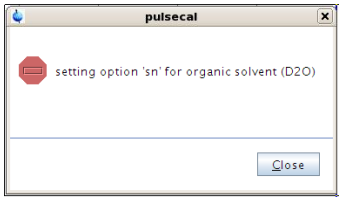
- Max positive signal = 90°
- Null points (no signal) = 180°, 360°

Note

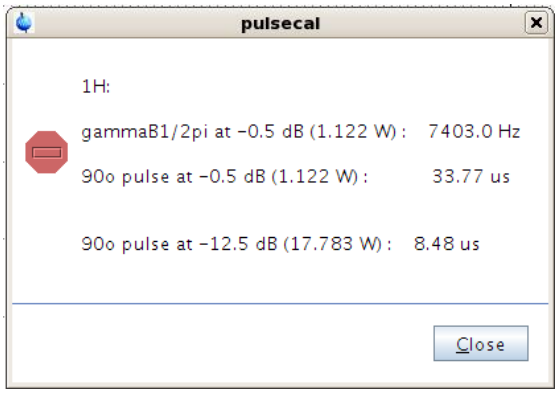
- Optimization of the 90° pulse width is critical in experiments, which involve many RF pulses.
- The solvent type influences the 90° pulse width. Samples in aqueous solvents will have 10% - 15% longer pulse widths. Increases in the ionic strength of the solvent, will increase the pulse width.
- The 90° pulse width depends on the power level at which it is applied. For BRUKER instruments, the 90° pulse power level refers to the attenuation of the maximum pulse power (db). Smaller values indicate stronger pulses.
- The method chosen for the 90° pulse width determination depends on the class of solvent used, protonated or deuterated.

2. Pulsecal – Fully Automated Pulse Calibration

- Make sure all RF-channels used are properly tuned and matched (*edasp, ATMM or ATMA*)
- Type the command *pulsecal* into the command line of the Topsin window and wait.
- Confirm the solvent by closing window
- Wait for measurement to take place



- A window with the pulse calibration information appears. Clicking on “Close” transfers the pulse calibrations into the current parameter set. In addition, all Proton Pulses of the current Bruker Standard Pulse Sequence will be re-calculated based on this new pulse determination and all X-pulses will be read in from the “prosol” table.



Remember: this calibration works only properly with Bruker Standard Pulse Sequences

3. Deuterated Solvents

3.1. Optimization of P1 using PAROPT or POPT (newer version)

3.1.1. The Experiment

- Set up a 1D proton experiment. (rpar AA_proton.MV, lock, tune, and shim)
- Select the getprosol icon  to update the default pulses, P1 and PL1.
- Change Pulse Program from zg30 to zg
- Set the following parameters: NS = 1 D1 = 4 P1 = 5
- Acquire and phase the peaks in the spectrum positive.

3.1.2. Edit the Display Region

- *This procedure is optional for manual calibration.*
- Select a region or single peak to display. Non solvent peaks are preferred.
- Using the cursors, select the region to display. (left mouse button)
- Update the window parameters using the right mouse button. (Fig. 2.1)
  - Select the first option "save display region to … "
  - In the next window, select "Parameters F1/2 …" and OK.

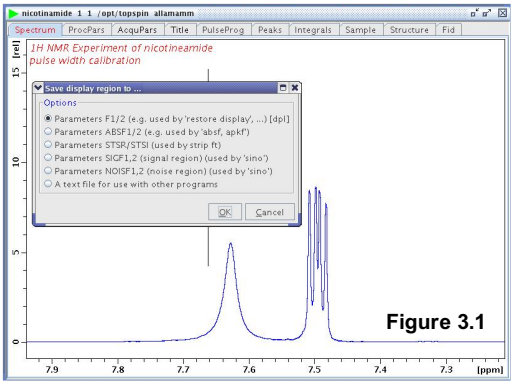


Figure 3.1

3.1.3. PAROPT or POPT for Parameter Optimization

- With the proper signal selected for display,
- Type "paropt" or "popt" and enter the parameters below.
  - Parameter to modify: p1
  - Initial parameter value: 5 us
  - Increment: 5 us
  - Number of experiments: 12
- DO NOT EXCEED a P1 value of a 450^{o} pulse.
- Type kill to stop the paropt process a few increments past the P1 (360^{o}). (Fig. 2.2)
  - Highlight the paropt process in the table.
  - Select KILL.
  - Enter OK to kill.
  - Repeat as necessary.

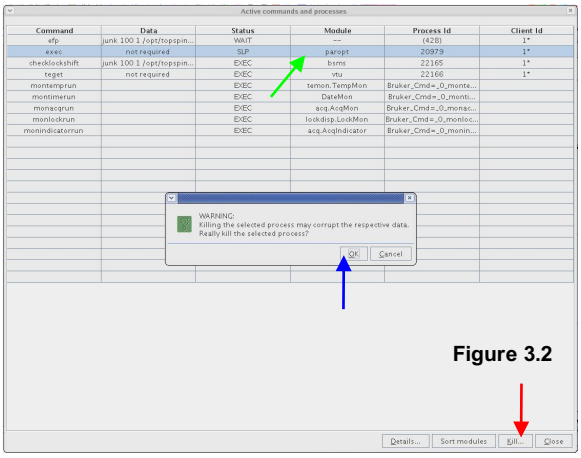


Figure 3.2

3.1.4. PAROPT P1 (360°) Arrayed Result

- Determine the value of P1 for the null by counting the number of peak groups displayed. (Fig. 2.3)
- Refine the result by decreasing the increment.
- Verify the P1 (360°) result is the null by acquiring a single scan, manually. (Fig. 2.4)
- Set P1 = P1 (360°)/4.
- **POPT** will calculate the value and display it in the title

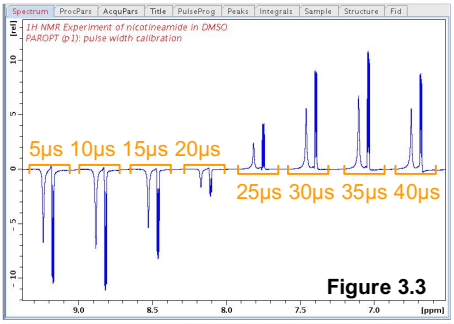


Figure 3.3

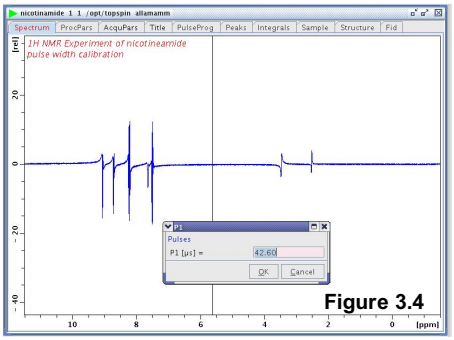


Figure 3.4

3.2. Manual Calibration of P1

3.2.1. The Experiment and Display Region Definition

- Set up a 1D proton experiment and display region as directed in 2.1.1 and 2.1.2.
- Change Pulse Program from zg30 to zg
- Set P1 to a value near P1 (270°) according to getprosol, or the CSB webpage for parameter settings. Run the experiment and phase the peaks negative. (Fig. 2.5)

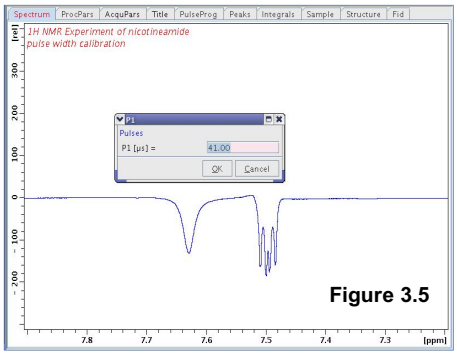


Figure 3.5

3.2.2. Coarse P1 (360°) Determination

- Repeat the experiment increasing the P1 value by 2 to 5 us. Process the spectrum using EFP. Continue to increase P1 until the signal inverts and appears upright. (Fig. 2.6)
- The value for P1 now exceeds the P1 (360°).
- Note the P1 value for which the signal inverts.

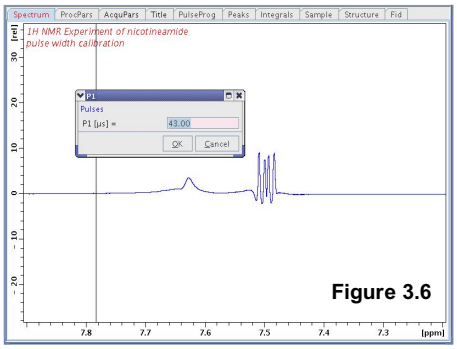


Figure 3.6

3.2.3. Refining P1 (360°)

- Refine P1 using 0.5 to 0.1 us increments. Repeat the experiment until the signal appears closest to zero intensity. Often a null will not be observed. Look for a spectrum,

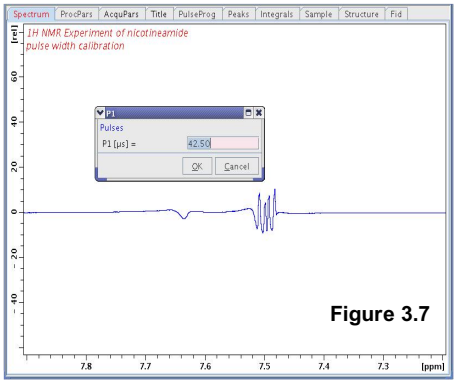


Figure 3.7

which is equally dispersive, meaning the peaks are balanced between positive and negative, or the integral is 0. (Fig. 2.7)

- Use P1 = P1 (360°)/4 at the PL1, power level used in this determination, for the next experiment.

4. Protonated Solvents

4.1. Optimization of P1 using GS

4.1.1. The Experiment setup

- Create a new experiment using the **A_pulse.MV** or **AA_pulse.MV** parameter set.
- Select the getprosol icon  to update the default pulses, P1 and PL1.

  The following parameters should be set at this point:

PULSEPROG	zg
TD	1024 or 1k
RG	1
D1	1sec
NS	1
O1P	4.7ppm
P1, PL1	according to getprosol, or the CSB webpage for parameter settings

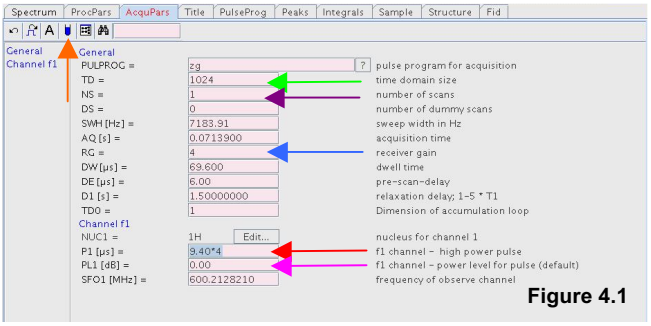


Figure 4.1

4.1.2. Optimizing the 90° Pulse

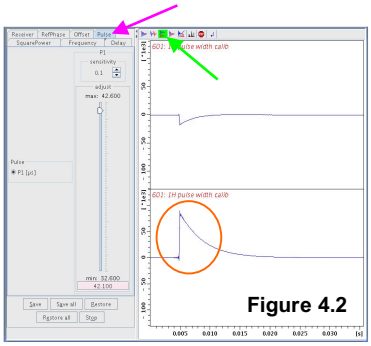


Figure 4.2

- Type "gs" at the command line.
- Display the real and imaginary FIDs separately. (Fig. 3.2)
- Select the Pulse tab.
- Note the phase of the pulse
- Change the pulse value to about 4x bigger then the current one since you are looking for a 360^0 Pulse.
- Adjust the P1 value by clicking on the scale, either above or below the slider until the initial points of the FIDs are minimized. The increment sensitivity might be changed with the sensitivity selector. Set the sensitivity to 0.1us for fine tuning.
- **DO NOT DRAG THE SLIDER. (Fig. 3.3)**
- If the first points in the FID don't show any intensity anymore you have found the P1 at 360°.
- Select SAVE or SAVE ALL and STOP.
- The 90^0 pulse will be P1 = P1 (360°)/4 at the PL1 used in this determination.

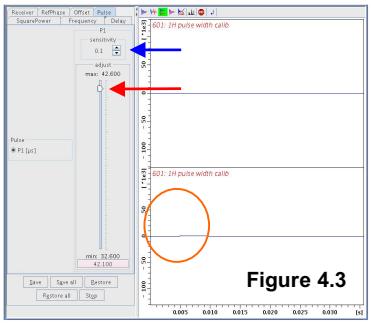


Figure 4.3

4.2. Note

Several additional parameters may be optimized with the GS subroutine as well:

- P [1…31] (**P**ulse width)
- PL [1…31] (**P**ower **L**evel)
- O [1…4] (Transmitter **O**ffset Frequency)
- SP [0...31] (**S**haped Pulse **P**ower)
- PHCOR [0...31] (**PH**ase **CORR**ection)