

Tuning, 90° Pulse Width Determination, and Temperature Control Instructions Varian VNMRS 500 MHz NMR

Probe Tuning

When: Before any experiment that requires an exact 90° pulse, for example, APT or a Gcosy.

Procedure:

1. Take a normal automated 1-D spectrum for the nucleus of interest. This first spectrum sets the appropriate experimental parameters for the observation frequency.
2. In VNMRS, pull down the Tools menu, slide right on Probe Tuning and choose Manual Tune Probe.
3. If you are not in the spectrum view mode click on the button at the top-left of the Autosampler page.
4. To tune the proton probe, choose channel 1. To tune the ¹³C or other low frequency nucleus, choose channel 2. Click on Start Probe Tune to plot the reflected power from the probe as a function of frequency. The goal of tuning is to minimize the reflected power at the operating frequency of the spectrometer. The operating frequency is denoted with a vertical blue (or magenta line) in the middle of the display.

For the proton side of the probe:

5. Find the knob on the left-most controller labeled “H-tune”, which controls the tuning capacitor. Note the position of the mark on the tuning knob. Draw the orientation in your lab book so that if something goes wrong you can return the knob to the starting position.
6. Adjust the tuning control to move the dip to the center of the vertical line on the computer display. Adjust the Matching control to give the lowest and sharpest dip possible.
7. On the computer display, at the right side in the middle, click on the Stop Probe Tune button.
8. Click on Quit.

Measuring the High-Power Proton 90° Pulse

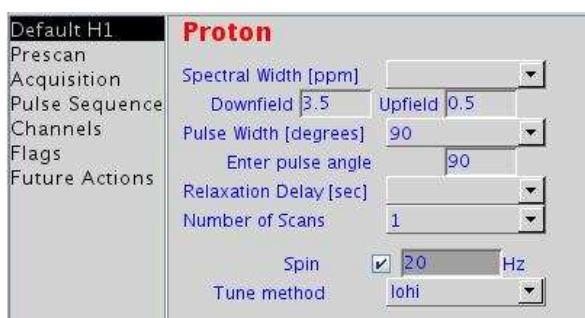
When: Before any experiment that requires an exact 90° pulse, for example, APT or a COSY.

Theory: A 90° pulse gives the biggest possible signal. However, adjusting the 90° width for maximum signal is difficult. It is far easier to adjust a 360° pulse to minimum signal and then divide the 360° pulse width by four to get the 90° pulse width. To determine an accurate pulse width you should wait $5 \times T_1$ between pulses to allow the system to return to equilibrium. Otherwise the signal will vary from pulse to pulse and you won't be sure if you have found the minimum signal. T_1 can approach 5 s, so the best delay is 25 s. However, using a 360° pulse allows you to cheat on this long time interval just a bit, since a 360° pulse returns the system pretty close to equilibrium after each pulse anyway.

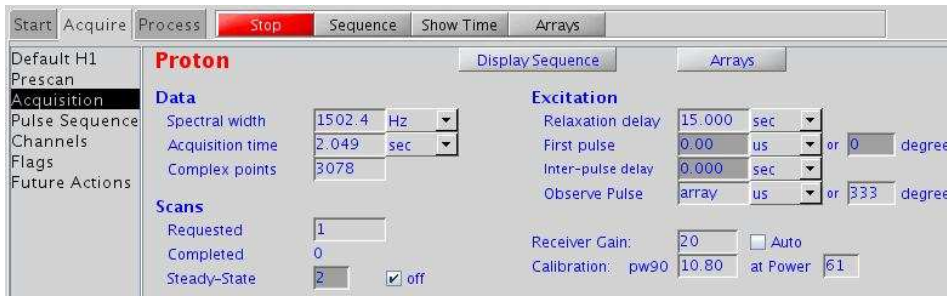
Use a non-sealed sample when measuring the pulse width to provide shorter T_1 values. A sample with a large sharp peak works best, for example, 20% normal CHCl_3 in D6-acetone. However, you usually just use your current sample, since you really want the 90° for your actual sample anyway. You will not be able to get zero signal for a sample with more than one peak. You just determine the best value you can.

Procedure:

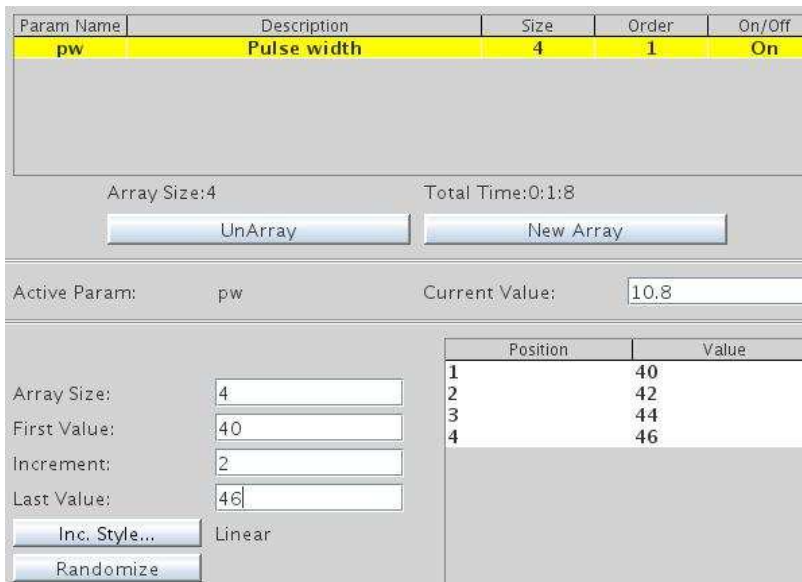
1. Tune the probe.
2. Expand the spectrum to focus on a small region of interest. Note the chemical shift values. For example, for ethylbenzene 3.5 to 0.5 ppm works well.
3. Set up another spectrum, however you need to enter the new spectral width before running the new spectrum using the following procedure. Choose the Proton experiment, as before. Choose the Acquire tab and choose the Default H1 page. In the acquisition parameters screen, enter the new chemical shift range. Set the Number of scans to 1.



4. We now need to set up a sequence of spectra with increasing pulse width: select the Acquisition page. Click off the Steady-state scans. Deselect Auto receiver gain. Set the relaxation delay to 15 sec:



5. Now we need to specify the pulse widths: click on the Arrays button. Enter pw in the Param Name box. Set the Array Size to 4 or more. A typical value for the First Value is 40 μ s and for the Increment is 2 sec or smaller. Make sure the chosen parameter values bracket the expected 360° pulse width.



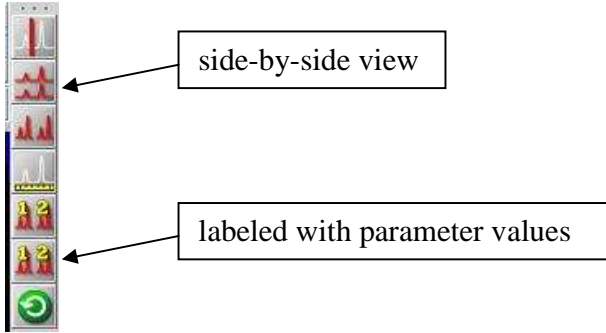
Click on Close for the Arrays dialog.

4. Check all your input values and make sure your sample number is highlighted on the Autosampler page. Now submit the run to the queue. When the run is complete, switch to the spectrum display screen.



5. Click on the Spectrum view button: →

6. Click on the side-by-side button in the new graphics tool bar as shown below. Also click on the “labeled with parameter values” button:



9. Find the value with the minimum intensity. Divide this value by 4.

10. Compare your 90° pulse to the value that is listed as $pw90$. If your value varies by more than a few microseconds, contact the system administrator.

Temperature Control

To determine the current temperature in the probe:

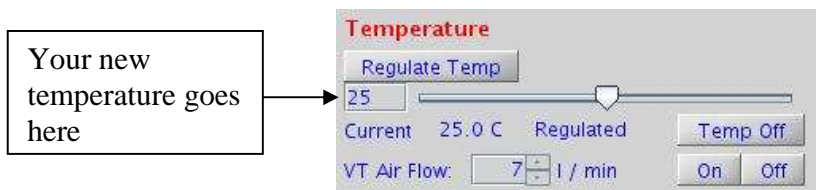
1. The current temperature is shown in the indicator box in the lower left-hand side of the VNMRJ screen.

Equilibrate at the new sample temperature before submitting your run to the sample queue.

Make sure to keep the temperature well below the boiling point of your solvent, or you risk a tube explosion in the probe.

To Control the temperature in the probe (above room temperature):

1. From the Start tab, select the Spin/Temp page. Normally, the VT Air Flow is set to 7 L/min:



2. Normally, the temperature will already be regulated as indicated by the “Regulated” indication to the right of the Current temperature setting. If already regulated, then all you need do is type in the new temperature in the dialog box to the left of the slider. If the temperature is not currently regulated, after typing in your new desired temperature, click the Regulate Temp button.

3. Let the temperature stabilize at the new temperature and then submit your sample to the queue.

4. When finished, **equilibrate the probe at 25°C** and then run ethylbenzene. Leave the temperature regulated at 25°C.

5. Contact the system administrator for temperatures below room temperature.