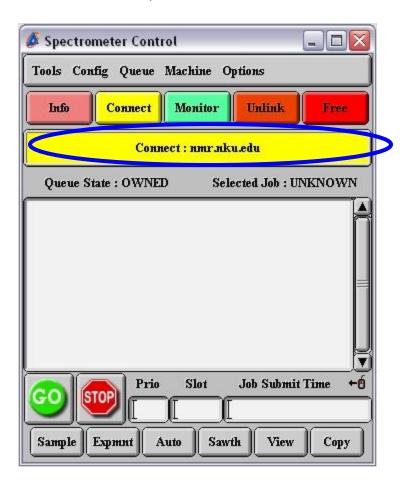
Illustrated NMR Guide

- 1. First start the Delta Software by clicking its icon beta found either on the Desktop or in the Start menu.
- 2. The window that appears is the main Delta window shown below. It will show important messages while you are running the software.

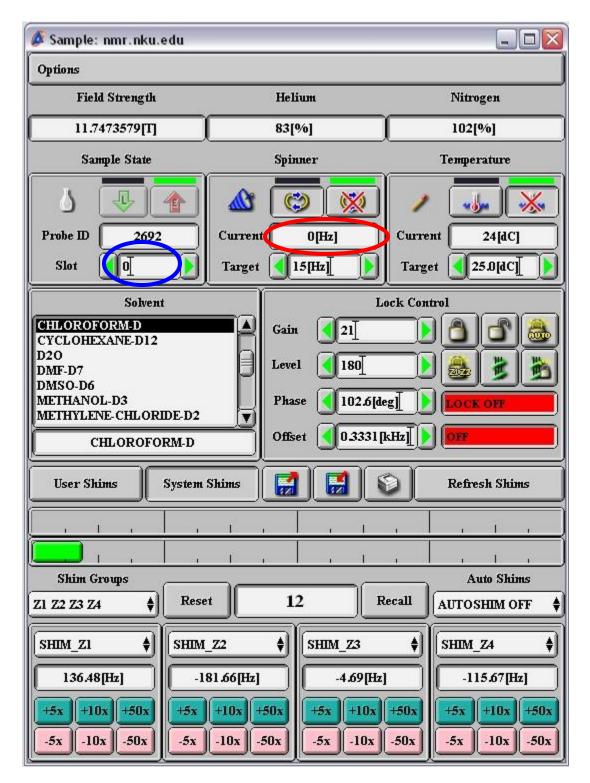
🖉 Delta	
File Processors Viewers Tools Analysis Acquisition	
Delta NMR Processing and Control Software Copyright 1990-2006 by JEOL USA, Inc. Version: 4.3.5 [Windows_NT] Network port = 6422	

3. From this window you can connect to the magnet by clicking the magnet icon (blue circle, see step 4) or manipulate data files by clicking the Data Processor icon (red circle, see step 10).

4. Clicking the magnet icon brings up the Spectrometer control window shown below. The software automatically connects to the magnet and the top portion of the window shows Connect: nmr.nku.edu and turns yellow (circled in blue). If it says anything else, you are not connected to the magnet typically because someone else is connected. You are not able to run experiments in this state.



This window also shows the experiment queue in the whitespace above the go and stop buttons, and gives you access to the Sample, Experiment, and Automation windows by the buttons along the bottom row. These windows will be discussed next. 5. Open the Sample window (shown below). This window contains a wealth of information about the sample inside the magnet.



First, to load your sample put the slot number into the field under the

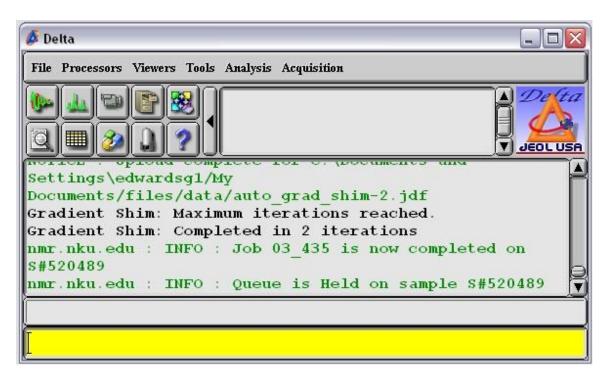
blue circle. The sample changer will automatically eject the sample that is inside the magnet (if there is one) and then pick up your sample/spinner combination and place it inside the magnet. While this is happening select the solvent that is inside your NMR tube. After your sample has loaded, watch the spin speed (red circle) to make sure the sample is spinning at the correct speed (someplace close to 15 Hz). <u>Trouble? Try ejecting your sample (by putting a zero in the sample slot) then reloading your sample. If this does not help try a different spinner, and then a different NMR tube.</u>

6. After the sample is spinning, select the solvent contained in your sample. We will lock onto the deuterated solvent signal by clicking the autolock button (red circle, as shown below) after a second or two the lock status will change to green and say lock on (just like the next picture).

<u>Trouble? If the spectrometer won't lock, make sure you have the</u> <u>correct deuterated solvent selected (you did use a deuterated solvent</u> <u>right)? If you are sure you are ok, load the system shims by pressing</u> <u>the button with the arrow pointing away from the disc. If this doesn't</u> <u>work find Dr. Edwards or your Advisor, we can fix it for you very</u> <u>quickly.</u>

Solvent	Lock Control
CHLOROFORM-D CYCLOHEXANE-D12 D2O DMF-D7 DMSO-D6 METHANOL-D3 METHYLENE-CHLORIDE-D2	Gain 25 Level 180 Phase 102.6[deg] LOCK ON
CHLOROFORM-D	Offset 0.3331 [kHz] [] [IILE
User Shims System Shims	Refresh Shims

7. Next click the gradient shim button (blue circle above). This will shim the magnet so that you achieve the best possible line width and shape in your resulting spectra. It takes a couple of minutes. When it is done, the main Delta window will show a message about being done in two iterations as shown below.



Trouble? Did you see an error message? Note it in the instrument log.

8. Now that the sample is loaded, locked, and shimmed. You are ready to run your experiments. Load the open experiment window (shown below) by clicking on the EXPMNT button in the spectrometer control window. You will now select the experiment of interest. (click on the global directory button in Red, then select your experiment single_pulse.exp for proton and single_pulse_dec.exp for carbon).

💋 Open Experiment		
Path: C: 🛊	C:\Program Files\JEOL\Delta\glo	bal/experiments/]
Format:	÷) Filter: (*.e	an I an
Directory	Filename	Version
- Favorites - Id Id_cosy Id_inadequate	 inept_dec.exp noesy.exp probe_tune.exp robot.exp roesy.exp sawtooth.exp single_pulse.exp single_pulse_dec.exp single_pulse_homo_gated 	
Ok Ind	b Delete Refresh	Cancel

This will bring up the Experiment Tool window for the specific experiment. I have shown the proton experiment below.

💰 Experiment Tool:	single_pulse.exp	
File Tools View O	ptions	
		Submit
Header Instrument Acquisition Pulse		
filename	[ld_spectrum]	Browse
sample_id	[
comment	Single Pulse Experiment	
process	(interactive_global 'std_proton_autophase.list';)	Edit
auto_gain	0	
force_tune	0	
		U
nmr.nku.edu To	otal Collection Time: 00:00:55	

Under the Header tab, you can change your filename and put in a sample identifier. I also suggest your click the auto_gain check box.

Under the Instrument tab, check to make sure the right solvent is selected, and changer_sample corresponds to the slot your sample was in. the recvr_gain will be set by the auto_gain routine later.

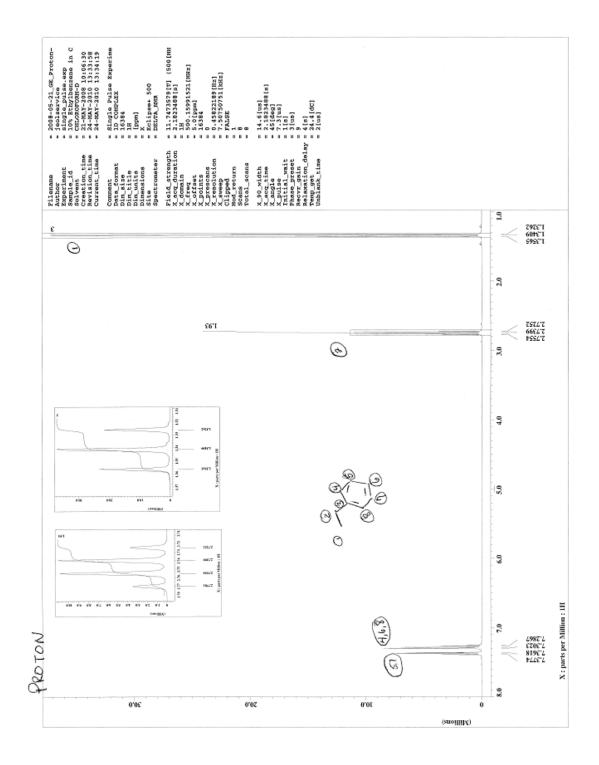
🎉 Experiment Tool:	single_pulse.exp	
File Tools View O	otions	
Header Inst	Tument Acquisition Pulse	Submit
solvent	CHLOROFORM-D CYCLOHEXANE-D12 D2O DMF-D7 DMSO-D6	
recvr_gain changer_sample		
nmr.nku.edu To	tal Collection Time: 00:00:55	

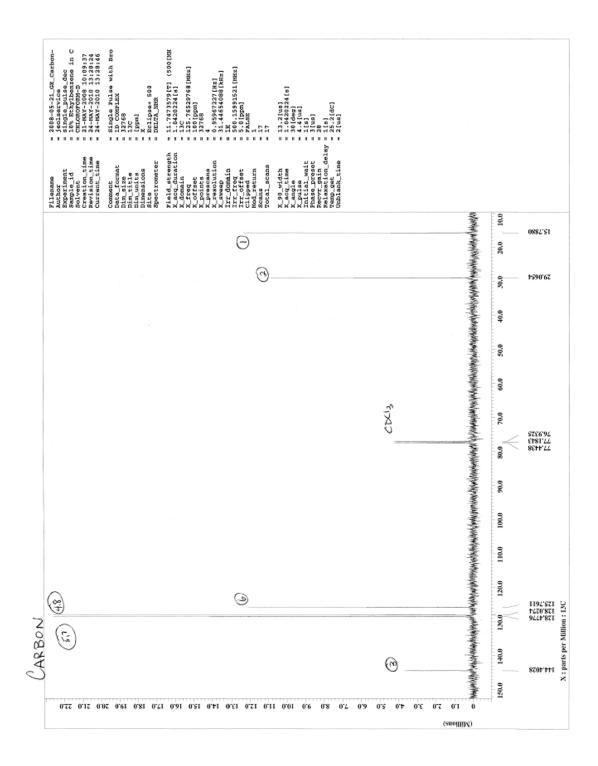
The acquisition tab has a number of instrumental features that you can change, but you really only need to be concerned with the number of scans. You can set this to any number you wish, larger scans will typically increase your signal to noise ratio, but also increase acquisition time.

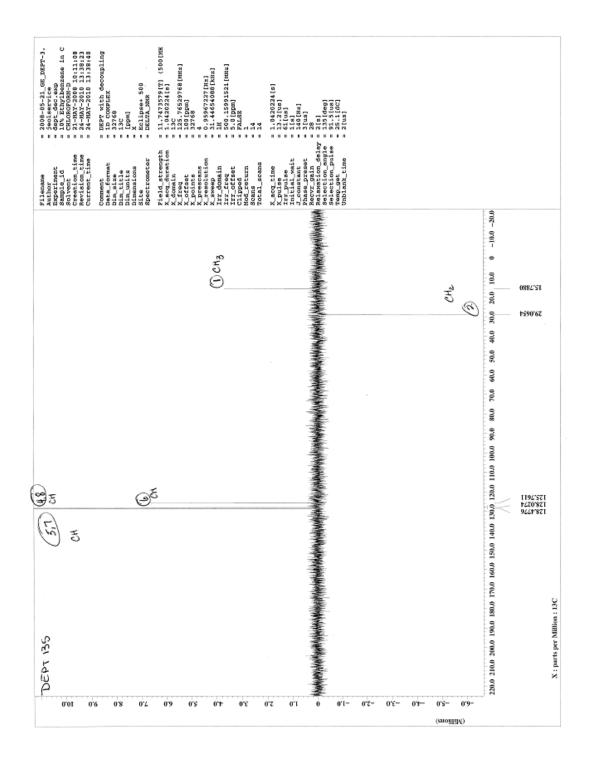
💰 Experiment Tool:	single_pulse.exp	_ 🗆 🛛	
File Tools View O	ptions		
	Add 💓 🛃	Submit	
Header Instrument Acquisition Pulse			
x_domain	Proton		
x_offset	[5[ppm]		
x_sweep	[15[ppm]		
x_points	16384		
scans	8		
x_prescans	I		
(mod_return	I		
x_acq_time	2.18383[s]		
x_resolution	0.45791[Hz]		
nmr.nku.edu Total Collection Time: 00:00:55			

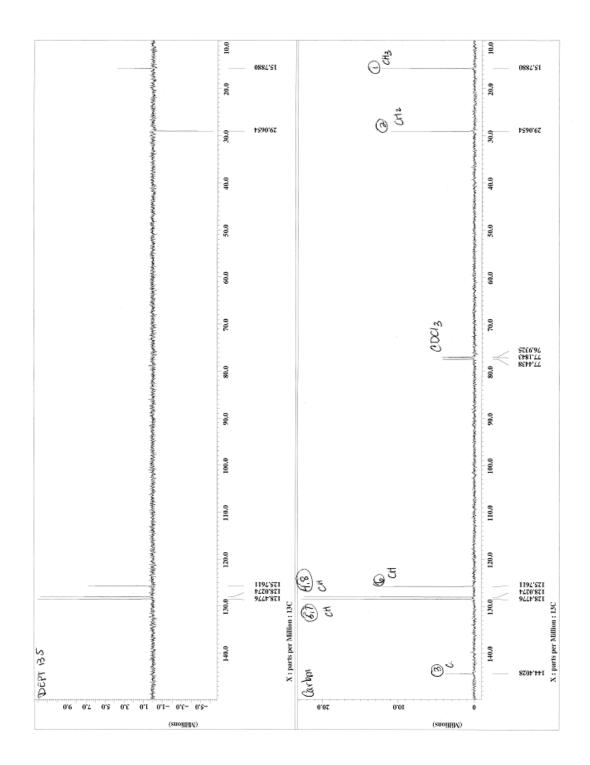
You do not need to mess with anything on the Pulse tab.

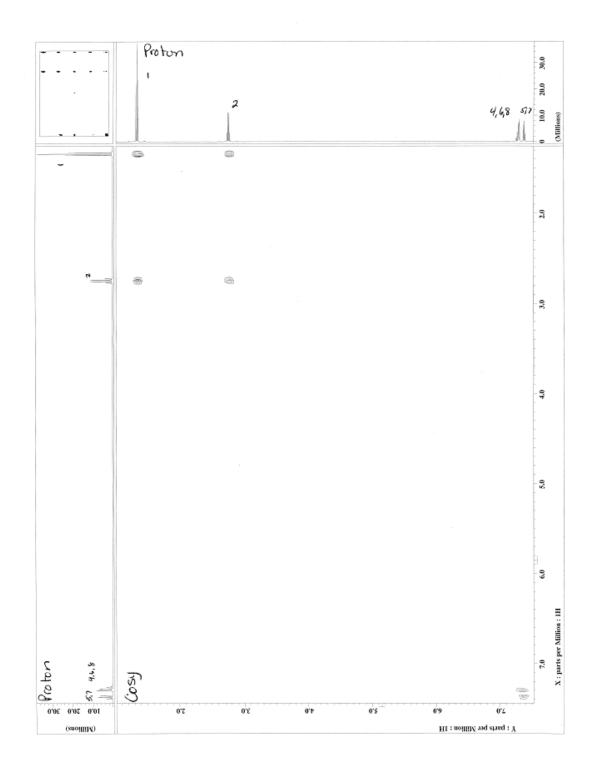
9. When you are done you can press submit and your job will be loaded into the Spectrometer Control window and the experiment will be executed. Once the job is complete, the data should appear on your screen. You can now eject your sample by putting a zero in the slot number of the sample window.











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