7. THE ACQUISITION PARAMETERS

We've arrived at the heart of the ^{1}H NMR demonstration lesson. We're on step #7 of our 1D acquisition and processing instructions list. This step will take us two videos to explain. In addition to a copy of the instructions, I recommend having a copy of the Basic Experimental Parameters nearby, which you can find on our website under the main menu link for Training.

Now we'll jump right in by clicking on the AcquPar tab.
The first parameter on the menu is the pulse program. This is a set of instructions telling the spectrometer when to wait, when to pulse, and when to acquire. The standard Bruker file named “zg” contains the spectrometer's instructions for the basic pulse-acquire NMR experiment. You should definitely watch the 5 minute youtube video by Lars Hanson entitled, “Simple demonstration of magnetic resonance as used in NMR and MRI” in order to understand how this works.

The parameter in the table, td, is the number of points used to digitize the FID. It is simply a number. It does not have any physical units.

The next parameter is ns, number of shots. This determines how many times the pulse-acquire experiment will loop, or in other words, how many FID's will be signal averaged to increase the signal-to-noise ratio. “ds” stands for “dummy shots” which is how many times we want to run the entire pulse-acquire experiment without saving the FID.

The spectral width in Hz is given by swh. If I want to know the spectral width in ppm, I need to divide by this parameter here, sfo1 – or I can let the computer do it for me by typing sw (without the h) in the command line. The current spectral width is set to approximately 20 ppm.

When I set the spectral width, I automatically set the digitization rate, since dw (the time between each of the td points when digitizing the FID, or the sampling rate or the dwell time) is equal to one over twice the spectral width in Hz. You can try it yourself: the inverse of our spectral width is one divided by 4000 and that gives 0.000250 (or 250 us), and half of that is 125 us, which is our dwell. This ensures that the Nyquist condition for digitization is met.

The next parameter is the acquisition time. This is the time that the detector is on, or the time during which the FID is being recorded. How long is that? It's the number of times the signal intensity is recorded multiplied by the time interval between each point. So, aq, our acquisition time, is equal to td (the number of points used to digitize the FID) multiplied by the dwell time, which is determined by one over twice the specified spectral width.

The receiver gain is how much to amplify the signal before digitizing it. The spectrometer will automatically determine the optimal receiver gain experimentally with the command rga. We'll right click on the “Acquisition information” window and select “Start rga.” This is step #7b) of our instructions list. How long it takes the computer to determine the best rg value depends on the parameters aq and d1. The bigger those values, the longer the rga routine will take. We can see what the automation routine is doing by looking at the blue lettered status report at the bottom of the screen. Once the program converges on an optimal amplification value, which depends on the intensity of the strongest signal in the sample, it will say “rga finished” and a new value for rg will appear in the table.

Next is dw, our dwell time, which is set by one over twice the spectral width in Hz. Right now it is 124.8 microseconds. That's the time interval between each of the 16 thousand data points which gives

us a total acquisition time of 2 seconds in order to record the FID.

The dead time, de, is usually 6 or 6.5 microseconds. The same coil is used for the transmitter and the receiver. After the rf pulse is turned off there is a spike called “ring-down” in the self-reonating circuit. We don't want to digitize the spike, so we'll throw out the first six or six-and-a-half microseconds before we begin digitizing the signal every 125 microseconds, or so.

The spectrometer can either pulse or not pulse before acquiring the FID. The pulse program we've requested tells the timing unit of the spectrometer to wait d1 seconds before the rf-pulse in our pulse-acquire experiment. D1 is the first delay. In this experiment it is the only delay. The difference between all the NMR experiments is in the placement, numbers, and durations of the pulses and delays.

In the margin here, it is written that we should set d1 from 1 to 5 times T_1. What it T_1? It is the relaxation time constant that explains the return of the nuclear spins to thermal equilibrium in the absence of an rf-pulse. T_1 is not a parameter we can change by altering any of the values in this table.

The nuclei behave like frictionless gyroscopes at the center of the atoms. Yet the turbulent molecular environment causes slight, but ever changing fluctuations of the the local direction and magnitude of the magnetic fields surrounding the nuclei. These continual magnetic “wanderings,” towards a steady-state orientation bias for the lower magnetic energy, depend on the time scales of the molecular motions of the surroundings with respect to the frequency of the transiently applied energy from the rf-pulse.

Each different proton in our ^{1}H NMR spectrum will have its own T_1 value. T_1. is also called the spin-lattice relaxation time constant or the longitudinal relaxation time constant (which are all synonyms). In short, I don't know the T_1 of this sample at this field without measuring it. For quantitative measurements, it is important to measure the longest T_1 value of the signals of interest, and to set aq + d1 $\geq$ 5 * T1. On the other hand, if you would like to suppress a peak using rf saturation, you should be pulsing faster than ~ one-half of T_1. For standard or “survey” ^{1}H NMR, a typical range of values are between 0-3 seconds when the FID is not being truncated. So, this default value of 2.5s is fine for now.

TDO is another loop that contains the ns loop inside it. Every ns the acquired data will be transferred from the CCU in the cabinet to this computer where I'm sitting. If something happens to the computer connections before the data is transferred, it will be lost. So, if I'm running a very long experiment, I might want transfer the data every so often and I can do this using TDO. Another strategy is to collect blocks of data in different experiment numbers using a command called multizg. If you use multizg, then at the end, you'll need to add all of the different experiments together.

The last parameters in the list are for the rf-pulse. With the zg pulse program, the shape is automatically set for a square pulse. P1 gives the duration of the pulse in microseconds, here it is 11 us. Pl1 gives the power of the pulse in terms of the attenuation (reduction) of the maximum power output. Here pl1 is -6 dB, which means that we'll be applying the maximum output power: a bit over 100 watts. The relative phase of the electromagnetic wave relative to a reference wave is defined in the pulse sequence itself with the parameters ph1 and ph31 for the transmitter and receiver phases, respectively.

Step #7c) is to start the acquisition of an FID using the default parameters for a single shot, which I'll do by clicking on this play button. Step #7d), where we tailor the acquisition parameters to this particular sample, based on the outcome of this scout shot, will be covered in the next video.