Changing Acquisition Parameters (For 1D NMR)

PART I – INTRODUCTION

1. For most routine experiments, the default parameters will work. However, you may encounter occasions in which the default parameters are not suitable or sufficient for your experiments.

2. This handout is intended to guide you through some of the more common parameters that are changed in NMR. It assumes basic familiarity with running a routine 1D NMR experiment.

Do all of the usual set-up stuff

1) You will need to do the usual things: log on, insert your sample, create a new experiment, read in the parameters, lock, shim, tune, etc.

Don't change anything you don't understand

2) When in doubt, do not change any acquisition parameter you do not understand.

PART II—CHANGING DATA ACQUISITION PARAMETERS

1. Read in default parameters for the nucleus that you intend to observe.

ased

1) Type **ased** to bring up a window of acquisition parameters. Select the parameter to be changed, type in the new value followed by Enter. To find a particular parameter within this menu, select the pink box next to the **Search parameter** "button" and type the parameter name. Another way to find a parameter is by selecting the appropriate category on the left side.

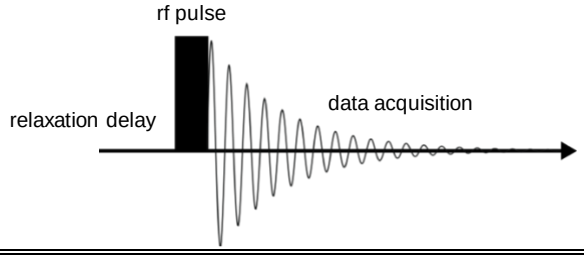
2) Do not change acquisition parameters that you do not understand.

3) If you know the name of a particular parameter, you can type it on the command line and enter the value. You do not have to use the menu.

solvent

4) The name of the solvent is a parameter. If you read in parameters with the **rpar** command after you have locked the sample, the **solvent** parameter will be overwritten. To correct it in the parameter set you are about to use to acquire data, go to **solvent** in the Lock section. Select the drop down button to the right of the current value and choose it from the menu that appears.

A. Routine data acquisition involves the following sequence:



pulprog

1) The name of the pulse program is the parameter **pulprog** and it is stored in the parameter files.

names of pulse programs

2) The routine sequence shown here is a pulse program called "zg" or a variation called "zg30" (see below). When the decoupler is turned on for broadbanded decoupling (as is usually the case for the observation of most nuclei other than ^{1}H), the pulse program is usually "zgpg" or "zgpg30" (see below).

relaxation delay

1) Relaxation decay (d1): a delay during which the relaxation of the spin system from the previous pulse occurs. The appropriate d1 value to use depends on the T1 relaxation time of the resonance(s) of interest and the length of other durations in the pulse sequence. In principle, to obtain the maximum amount of signal per scan, you should use a 90_o (□/2) pulse, and (d1+aq) ≥ 5* T1 of the slowest relaxing resonance, in which aq is the acquisition time (see below). However, when many scans are required, it a better use of time to use 30_o pulse and wait approximately 1*T1.

d1

a) In the default parameter files, **d1** is the relaxation delay. In most cases it is arbitrarily set to 2 seconds. (This is often too short for carbonyls or other non-protonated carbons! If you have a degassed sample, this is often much too short!)

pulse times & power levels

2) Radiofrequency (rf) pulse: this is a short, powerful rf pulse at the center of the spectrum. The pulse duration (p1) is given in microseconds. The pulse angle is directly proportional to the ratio of the pulse time used divided by the 90_o pulse time. In the literature, pulse angles are usually reported rather than pulse times, because the appropriate values are dependent on the hardware of a given system

p1

a) In the default parameter files, **p1** is the rf pulse time. It is set to a 90_o pulse. However, the name of the current pulse program is zg30 for a proton experiment, and zgpg30 for the default files for nuclei other than $_1H$. These pulse programs multiply **p1** by 0.33. Thus, a 30_o degree pulse is used in all the default parameter files.

getprosol

b) Type **getprosol** to ensure that the most recently determined values for pulse times and power levels for all nuclei in the particular pulse program will be used. Alternatively, in the menu of acquisition parameters, you can select the **Set probe/solvent dependent parameters** "button" (2_{nd} "button" to the left of the Search parameter "button").

Data acquisition

3) Data acquisition: the collection of individual data points that make up the nuclear resonance free induction decay (FID). The data rate = 2*sw. The time required to acquire each data point (dw) = 1/(2*sw). The acquisition

Relations among spectral width, data points, and acquisition time

time (aq) is equal to the time required for each individual data point (dw) times the number of data points acquired (td).

$$aq\ [seconds] = \left(\frac{1}{swh\ [Hertz]}\right) * \frac{td}{2}$$

aq, sw, and td are not independent parameters. Changing sw will change aq and keep td constant. Changing aq or td will change each other while holding sw constant. Ideally, you should match the acquisition time to the time when the observed NMR signal disappears into the noise.

ALWAYS check your aq if you change your sw

WARNING: Please pay attention when changing the sw for any experiment with decoupling. It is very easy to double or quadruple the aq, which could burn out the probe from high-power decoupling. If you have questions about this, please ask one of the NMR staff!

SNR

4) The number of times you loop through the entire sequence is called the number of scans. Provided that a resonance is not saturated, the signal to noise ratio (SNR) scales as √n. Phase cycling is carried out to minimize hardware-related artifacts.

a) For routine data acquisition, we suggest using **ns** as a multiple of 8 (the default value).

set td0 to acquire more averages

b) For signal averaging, please change **td0** to whatever value you need.

c) The total number of averages acquired will be ns * td0.

B. Based on your chemistry you may need to change:

1) The position of the center of the spectrum

a) Since the lock channel uses the same field value for all solvents, the center frequency is not dependent on the solvent. The center frequencies can be input in ppm.

o1p

b) The center of the observed spectrum is set with the parameter **o1p** (in ppm).

o2p

c) The center of the decoupling when observing nuclei other than ^{1}H is **o2p** (in ppm).

2) The spectral width (SW)

sw

swh

a) The ideal spectral width varies from sample to sample. We recommend that you start with a large spectral width because if it is too small or not centered properly you may miss peaks. The default parameter files have large spectral widths. The spectral width

ALWAYS check aq if you change sw

parameter is **sw** (in ppm) or **swh** (in Hz). Don't forget that changing the spectral width will change **aq** as discussed above.

WARNING: Please pay attention when changing the sw for any experiment with decoupling. It is very easy to double or quadruple the aq, which could burn out the probe from high-power decoupling. If you have questions about this, please ask one of the NMR staff!

2. Strategies for long term data acquisition can be used to efficiently utilize spectrometer time.

a) Set **ns** 8. Set **td0** to a number larger than 1 (e.g., 128). Start data acquisition with **zg**.

tr

b) To check the progress of the data acquisition, type **tr**. This will transfer the data in the acquisition memory and add it to the disk file identified by your current data set name and expno. You can then process the data as usual while the acquisition continues. You can repeat this process as long as needed to obtain the desired signal to noise ratio.

c) The **tr** command will add the data to the data previously stored with **tr** and zero the acquisition memory. The signal displayed in the acquisition window is only the result of data accumulation since the last **tr** command.

halt

d) When data acquisition has proceeded long enough, stop it by typing **halt**. Remember that the signal to noise ratio increases as the square root of the number of scans (provided that the resonance is not saturated).

Status parameters button

e) Whenever an acquisition has been halted prior to completing **ns** scans, in the **AcquPars** tab menu, select the **Status parameters** "button" to see how many scans were acquired along with all other acquisition parameters that were used to acquire the stored data. (These parameters can be different from those seen initially, which are the parameters that will be used to start the next acquisition.)