

Measuring simple 1st order kinetics with the Varian

All commands written in **bold type** are direct input at the input window prompt or are a standard VNMR button. Note that kinetics measurements (using the **kind**, **kinds**, **kini**, or **kinis** macros) are available on all of the Facility Varian NMR spectrometers.

This tutorial will show the user how to setup an experimental array using the Varian parameter '**pad**'. **pad** allows the user to specify a predetermined delay between sequential acquisitions. Once completed, the data set is then fit to either an increasing ($I = I_0 e^{-kt}$) or decreasing ($I = -I_0 e^{-kt}$) exponential function. Remember that in order for either of these kinetic analyses to be valid, the experiment must be setup to emulate pseudo-first-order kinetics, but of course you knew that.

Setup and data acquisition:

- Log onto the spectrometer and insert your sample into the magnet in the usual fashion.
- Choose the desired nucleus and solvent in the usual fashion. You may spin the sample but it is not necessary.
- Set the temperature to 20°C or whatever temperature you wish to perform your experiment. Remember that a stable sample temperature is an important factor in kinetics acquisition and analysis. If, for example, you wish to do your experiment at 20°C:
 - Type **temp=20 su**. If this doesn't work do the next step.
 - Type **temp**. In the Temperature Control Window click the **[Reset VT]** button. You may also select the 'Turn temperature control on at ...' radio button and use the slider bar to select the desired temperature.
 - If you continue to have temperature stabilization problems see our VT problems page or contact a staff member.
- Lock the sample and check for lock power saturation. Do not saturate the lock channel.
- Tune the probe. Skip this step if using the Mercury 300.
- Shim the sample. If you are not spinning the sample you may wish to 'touch up' the x-y shims.
- Run a normal 1D proton spectra with a reduced **nt**. Choose the desired spectral width using the cursors and press the **[Expand]** button. Type **movesw ga** and then wait for the experiment to complete.
- Now, we are ready to setup the experiment. Suppose you wish to acquire a 64 scan proton spectra every 15 minutes for 6 hours. Ultimately, you will end up with 25 data sets from which to perform your kinetic analysis. These parameters will be used in the following example. The experiment can be setup as follows:
 - Type **nt=64 time**. A message similar to "Total acquisition time is 3 minutes 19 seconds" will appear in the Status Window. Therefore, EACH acquisition of 64 transients will take 199 seconds. Time will vary slightly from instrument to instrument.
 - Now setup the array. Type **array** in the Input Window. The query "parameter to be arrayed:" will be displayed. Respond with **pad**. The acronym stands for **pre-acquisition**

delay. You will now be asked “enter number of steps in the array:”. Using our above example type **24**. You will now be asked to “enter starting value:”. Enter **701**. This is 15 minutes (900 seconds) minus 199 seconds (the total acquisition time). You will finally be asked to “enter array increment:”. Enter **0**. This will setup the array such that the time between acquisitions remains constant.

- The array values will automatically be displayed in the dg interface. Finally, you must change the first array value from 701 to 0. This will start the experiment immediately rather than delay the initial start by 701 seconds. To do this type **pad[1]=0 da**. This will redisplay the array with the new pad[1] value of 0.
- The array may also be setup by typing **pad = 0,701,701,701,...** etc. 20 more times.
- Type **gain?** Set the gain to the value returned in the Status Window (for example 20) by typing **gain=20**.
- Start the experiment. Type **au** (submit experiment to acquisition and process data).
 - You can view the experiment progress by typing **ds(1) vsadj wft**. (Display 1st spectrum (array index 1), Automatic vertical scale adjust, Weight and Fourier transform 1D data).
 - If you wish to view the data during the acquisition type **wft dssh**. This will perform the weighted Fourier transform on the completed transients and **display the spectra stacked horizontally**.

Processing the data:

This section will guide you through the processing portion of the experiment by using the above example as the guide.

- In our case, arraydim=24, (array dimension) type **wft(25) ds**. This will perform a weighted Fourier transform of the last spectrum acquired. If the desired peak has decayed into the baseline, you should type **wft dssh** and select a suitable spectrum.
- Phase the spectrum properly then select a threshold by clicking the **[th]** menu button and selecting the desired threshold using the left mouse button.
- Type **dll fp**. This will display an indexed line list in the Text Window and measure the peak heights for each peak in the array of spectra. You may also type **ll** to send an indexed line list to the printer. We suggest a hard copy to make the selection process easier.
- Depending on the type of kinetics analysis required, type
 - **kind** Kinetics analysis, decreasing intensity
 - **kinds** Kinetics analysis, decreasing intensity, short form
 - **kini** Kinetics analysis, increasing intensity
 - **kinis** Kinetics analysis, increasing intensity, short form
 - Details for each of these macros may be found in the Command and Parameter Reference.
- Type **expl** to display the kinetic fit. Type **pexpl** to plot the fit.
 - Again, more details for each of these macros may be found in the Command and Parameter Reference.