Kinetics experiments with acquisition of two nuclei

Eugenio Alvarado.
Department of Chemistry, University of Michigan. 02/09/11

In a kinetics experiment, a series of 1D spectra is acquired where the spectra are recorded at regular intervals of time. This is accomplished by delaying the acquisitions with a "pre-acquisition delay" (`pad`) after the previous spectrum was recorded. It should be possible, with a sufficiently long delay, to record the spectrum of a second nucleus during this time. In this way, it should be possible to follow a kinetics experiment by observing two nuclei, for example ^{1}H and ^{31}P spectra. The macro `UMkin2nuc` can be used to set up such experiment easily.

The acquisition parameters for the two nuclei should be set up in workspaces 1 and 2 (exp1 and exp2). The `UMkin2nuc` macro will then set up the acquisitions in workspaces 1001, 1002, 1003, etc for the first nucleus and 2001, 2002, etc. for the second nucleus. The acquisitions will be done in the order 1001-2001-pad-1002-2002-pad-1003-2003-etc. The sequence of events will be:

--nuc1[1]-nuc2[1]---pad--nuc1[2]-nuc2[2]---pad---nuc1[3]-nuc2[3]---...

After the setup, typing `au` (do not use `go` or `ga`) in experiment 1001 will start the chain of acquisitions.

Procedure:

1. In workspace 1, load parameters for the acquisition of nucleus 1. Change the parameters you want including `sw`, `nt`, temperature, etc. Read the notes below.
2. In workspace 2, load parameters for the acquisition of nucleus 2. Change the parameters you want including `sw`, `nt`, temperature, etc.
3. Use `UMkin2nuc` to set up the acquisition. The macro will ask the number of desired spectra in each series and the repetition rate in seconds (the time between the start of two consecutive spectra in each series).
4. Type `au` to start the acquisitions.
5. When the acquisitions finish, use `UMarrayfids` twice to group the individual fids of each series into a single arrayed fid. The macro will ask the workspace number to start from (enter 1001 or 2001), the number of fids, the parameter to array (always `pad`) and a file name for the result. The resulting file can be processed as usual. To see the `end_time` of a particular spectrum in the array type `write('line3', end_time[`***n***`])` where ***n*** is the number of the spectrum. If you use `UMdli` to get a list of peak integrals, the values of `end_time` will be listed too.
6. `UMrmexp` can be used to remove the unneeded workspaces 1001 and above.

Notes:

1. Make sure the probe is tuned to both nuclei, and the temperature has reached equilibrium and is set to the same value in both exp1 and exp2.
2. Autolock and autoshim should be off.

3. The acquisition of the second nucleus must fit inside the time between two consecutive acquisitions in the first series, or you will get an error message.
4. Don't use vnmrj's series of analysis macros `kind`, `kinds`, `kini` and `kinis`. They do not consider the time spent in steady state scans and can make inaccurate calculations leading to wrong results.
5. The *actual* end time of each spectrum (fid) will be stored in the parameter `end_time`. Its value can be displayed in vnmrj's command line with: `write('line3', end_time)`. The end time is stored as a Unix timestamp, which is the number of seconds past since Jan 1, 1970. For your information or curiosity, this number can be converted to a human readable date with the following command in a Terminal Window (replace the *number* with the value of `end_time`):
   `date -d @`*`1296513572`*
6. Use the actual times in `end_time` for your analysis. Times calculated from parameters like `pad`, `nt`, `d1` and `at` may not be very accurate because of unpredictable delays needed to switch among workspaces and to change the hardware (like the transmitter nucleus), and the errors will add up and become more important for the last spectra in the sequence.