This document describes how to acquire $T_1$ data, $T_2$ data, and kinetics data as well as some details on how to process the data. Processing using the Bruker Topspin module Dynamics Center is discussed. You could also consider running your own processing for more flexibility.

**T1 Data Acquisition**

- Be sure shimming, atma & rga are optimized.
- Run a standard 1D proton spectrum of your sample.
- Both $T_1$ and $T_2$ experiments require a relative accurate 90-deg pulse length. You will need to calibrate it for each sample. Refer to my blog for procedures.
- Type `edc`. In the new file window, click *Experiment*, select *T1IR*. Click *Set Selected Item in Editor*. Check *Execute “getprosol”*. In the *Title* window, write down date; sample info; temperature of the expt; etc. This experiment method is called ‘Inversion Recovery’ and involves a 180 degree pulse followed by a delay (which is given a series of values) and then a 90 degree pulse. During the delay period the magnetism relaxes toward equilibrium.
- Type `ased`. The window that comes up contains all the parameters needed to run this experiment. In P1 blank, input the value that you determined (usually 11-13 us; if you determined a value that is very different from this, don’t proceed! Let me know – something must be wrong). VDLIST contains the variable delay period between the 180deg and 90deg pulses. You can click the *E* button to view the list. You could also edit the list and customize your experiment.

Here is an example of the VDLIST:

1. $u$
2. $50m$
3. $100m$
4. $200m$
5. $300m$
6. $400m$
7. $500m$
8. $1s$
9. $2s$
10. $5s$

Note that there are 10 values and that the designations $u$, $m$ & $s$ are crucial!!! You are not restricted to 10 values. Without $u$, $m$, or $s$ the default will be seconds.

The complete recycle delay ($d_1 + aq$) must be at least 5*$T_1$. In our T1IR experiment, I set the default $d_1$ to be 15 s and $aq$ to be 0.5 s. This should be long enough for most molecules.
You may now acquire data. To estimate the time required, enter the command *expt*.

During the experiment, after a couple of slices have been acquired (check the status bar for info), you can type *xf2* and do phase correction for the slices that already have been acquired. The phase correction that you apply will be saved. When the experiment is done, type *xf2* again, and the phase correction that you did for the first few slices will be applied to all the slices. Use *Multiple Display* to check that all the slices indeed have good phase correction. Refer to the last section of this handout for more details on data processing.

**T2 Data Acquisition**

Run a standard 1D proton spectrum of your sample. Be sure shimming, atma & rga are optimized.

*edc* the standard proton file to a new name.

Change the pulse program:

*pulprog cpmg*

change the *parmod* of the file to 2D :

*eda* and then select 2D from the pull-down menu on that parameter slot (accept any questions asked about deleting sub-files)

This cpmg pulse program (*Carr,Purcell, Meiboom, &Gill* contributed to its development over a number of years of NMR evolution) involves a 90 degree pulse followed by a series of C 180 degree refocusing pulses in the x,y plane spaced by a period, 2*d20*, which determines how long the magnetism has to relax, i.e. 2*C*d20 before acquisition.

Create a vc list:

*edlist vc* and enter a ‘new list name’ such as t2-sample.

On the window that appears enter at least 8 values of c. d20 should be *at least 50*P2 or a minimum of about 1millisecond (1m).

A good starting value might be d20 10ms. (In some multi-environment regimes the value of T2 is a function of d20!)

set d20 value:

*d20 10m*

One might enter the following values of C:

4
8
20
For each C, the delay (i.e. the x coordinate) is 2*C*d20. With d20=10ms, the longest delay in the above list is 10ms * 2 * 160 = 3.2 s

Even values of C are chosen so that 180° refocusing pulse errors cancel. Choose NS 8*n so that phase cycling also cancels out receiver errors.

The fitting programs will be more reliable if the T2 relaxation is taken to an intensity of zero at the longest delay, but this may require some trial and error with the VCLIST values and d20. For signals with short T2 (e.g., < 100 ms), you should consider use a shorter d20, e.g., 2 ms. Usually, small molecules (MW < 1000) have T2 of > 1 s, while larger molecules and aggregates have shorter T2. When the aggregate is very large (e.g. > 100 nm), its signal will vanish since T2 is so short and thus the peak so broad that the signal is in the baseline.

You need to be sure that between each C value the system starts back at equilibrium. So set d1+aq >= 5*T1.

You can estimate the total length of the experiment with expt.

**Acquiring Kinetics Data on Bruker 500**

1. Create a new file with edc. In edc window, click Experiment, and choose KINETICS1H (or KINETICS31P – simple 31P without 1H decoupling). check getprosol.
2. Enter d20 and input your desired experiment interval in seconds (eg. 60)
3. Enter td and input your desired number of experiments (slices) in the second blank. It should be <= 16. (It's possible to increase this. First type si and give it a bigger number in the second blank, e.g. 32. Then you can increase td to a number that is <= si).
4. Enter desired ns and ds (dummy scans). (ns+ds)*(d1+aq) should be <= d20 so that your sample is not in a transient state within the same slice. If you have enough signal, it is strongly recommended that you use ns of 1 and ds of 0.
5. Type in expt and see if the projected experimental time agrees with your estimate. If not, something must be wrong and please go back to check all the parameters.
6. The data will be in 2d format. Type xf2 when a few slices have been acquired to make sure that things are in order.
**Processing Data**
Each of the above types of experiment is saved as ‘serial’ files, or pseudo-2D files. This means there are a series of 1d fids stored but only one set of parameters. All of the types of data are processed in very similar ways. This is treated as though it is 2d data even though it is really just a series of 1d spectra.

To convert serial fids to spectra: \( xf2 \) (Fourier transform in 2\(^{nd} \) dimension)

The resulting display is a color-coded intensity plot as you look down on the series of 1d spectra stacked in the y direction. Use *Multiple Display* to view all the slices. The first spectrum is at the bottom of the ‘2d’ display. By default, negative intensities are green and positive intensities are blue. This is important for inversion recovery T1 data where the first spectra should be negative. For T2 and kinetics data peaks should all be positive. Increase the peak intensities a lot to check the baseline, and determine if phase correction is needed.

*Phasing the 1d spectra*
Please refer to my blog for phase correction. For kinetics and T2 data spectra would be phased for all positive. For inversion recovery T1 the first slice should be phased negative and hopefully the last slices phased with the same parameters will be positive.

*Baseline correction* is usually necessary. Please refer to my blog.

*Dynamics Center*
You are now ready to enter the Bruker kinetic data processing module. Click on Analysis at top of window. Click on *T1/T2 Relaxation, then Dynamics Center*

Follow the flow of the direction. My blog has an entry about Dynamics Center: http://blogs.umass.edu/weiguoh/?p=713

*Do it yourself one by one:*
Some times it might be advantageous to split a 2D file into a series of 1D files: type *xau splitser*. This will allow you to manually process each individual slice.