Measurement of T₂ Values

The T2 relaxation time of NMR peaks may be measured through the Carr-Purcell-Meiboom-Gill (CPMG) pulse sequence,



which prevents the *z*-magnetization from relaxing while the transverse magnetization undergoes coherence loss. This sequence is repeated for a series of loop counts (n), and the resulting signal intensity is regressed *vs.* the total delay time.

Setup/Acquisition

- 1. Inject a sample, lock, shim, and acquire a proton spectrum as normal, without spinning (ro off).
- 2. Change pulprog to cpmg. Select the AcquPars tab (or type eda) and use the $\downarrow_{1,2,..}^{\downarrow}$ button to convert the parameters to 2D.
- 3. Create the vc-list, which contains the *n*-values to be used, and set the vclist parameter:
 - edlist vc t2delay
 - vclist t2delay
- 4. Adjust other parameters for the CPMG sequence:

d1	$= 5 \times T_1$ for ¹ H	typically 20 s; long is critical
td(F1) = number of items in vc-list		
d20	= 10m	relaxation delay $(\frac{\delta}{2})$
ns	= 8	a multiple of 8
ds	= 2 or 4	

5. Start the acquisition (rga; zg). Type expt to see how long the experiment will run — usually about 40 min.

Processing

- 6. Set si(F1) to a power of 2 greater than td(F1). Set the F2 line broadening, 1b(F2), to 1.
- 7. Type xf2 to Fourier transform data-set.
- 8. Enter "rser 1" to select the first slice of the spectrum in a new window. Type efp to Fourier transform the slice.
 - a. Expand on the region of interest.
 - b. Phase the spectrum. Ensure that the baseline looks good and multiplets are symmetric, even if the multiplets do not properly phase.
 - c. Integrate the peaks of interest:
 - Type .int and integrate each peak for which T2 should be calculated.
 - Save as "intrng" and return, using the A and buttons.
 - Type wmisc intrng t2rng to write the integration-region list as "t2rng".
 - d. Set the frequencies for which T₂ will be calculated. As you do this, it is important to only choose *one* line within each integral region, and to choose the line with the maximum intensity.
 - Type .bas1 to enter baseline-correction mode.
 - Use the \perp button to pick baseline points.

- Save and return. The software will complain about insufficient points. You can verify that the points are indeed picked by clicking $\frac{1}{12}$ again: red boxes with arrows should appear on the peaks. Click cancel.
- Return out of the peak-pick subroutine.
- Type wmisc baslpnts t2bas to write the baseline-points file as "t2bas".
- e. Create a delay list from your vc-list (loop-count list).
 - Open the terminal emulator (right-click on desktop, choose "Konsole") and, at the command prompt, enter vc2vd.
 - Enter the acquisition parameters it asks for (d20, p2, and vclist) from the acquisition parameters (ased) or pulse sequence (spdisp).
 - Write the new vdlist to t2delay.
- 9. Return back to the 2D spectrum and enter proc_t1 into the TopSpin command line. The macro will ask for a series of parameters. Reply with the following:

FID number =	1	
limits =	1000 to -1000	
drift =	20	
baselinepoints file =	t2bas	
integrals file =	t2rng	
vd file =	t2delay	(or other filename chosen through vc2vd)
calculation type =	2	

10. Once the calculations have been performed, obtain the results by clicking the Analyse : Dynamics :

T1/T2 : Relaxation buttons from the top tabs. If any bad data-points need to be omitted,

- a. Click the check-list icon to verify that the Function Type is UXNMRT2.
- b. Click the +/– buttons to select the peak you wish to modify.
- c. Click the $|\downarrow|$ button, move the cursor to select a data-point, then right-click on it.
- d. Once the point is deleted, the (>) and (\gg) buttons will recalculate the fit.