

T1 Measurement using Topspin

Background

Attached to this handout is a discussion of T_1 relaxation and its measurement taken from R. Freeman, *Handbook of NMR*. Read this before proceeding. The basic sequence for measuring T_1 is the inversion-recovery sequence. This is a non-selective experiment in which all peaks are inverted and measured simultaneously. (This has implications for the T_1 measurement of coupled proton spectra). The sequence is:

180 - tau -90

This sequence is repeated for a series of delays, tau, during which relaxation occurs. For each tau value a 1D spectrum is taken. The intensity of peaks in these spectra, when plotted against tau, describe the relaxation (a recovery curve).

The software treats the T_1 experiment as a pseudo-2D experiment. When doing a T_1 experiment, the 2D window will be present. It is actually an arrayed 1D experiment but that is very similar to a 2D experiment. One can look at the individual 1D spectra using the 2D commands, rser and rsr, for reading in "slices".

Setup and acquisition

Type rpar t1ir.top to read in the T_1 measurement program and parameters.

Variable delay list

The first thing that must be done is to create the list of variable delays. Type edlist, select variable delay (vd), and create a new list or chose one of the existing lists. (You can only make changes to the lists that you have created yourself). At least 10-15 delay values are needed to accurately describe the recovery curve. The delay values should be exponentially spaced for maximum accuracy. The choices of delay depend on the value of T_1 . One value should be 4 or 5 times T_1 , which will represent the fully relaxed spectrum. See the examples at the end of this handout. The following parameters must be set and are found in AcqPars:

vdlist - must be set to the name of the list you created above

td in fl - must set to the number of entries in the vdlist

ns - must be a multiple of 2

d1 - relaxation delay -should be 4 or 5 times T_1 .




sw, o1p, - these and any other standard parameters should be set for your particular spectrum




Type expt to see how much time the experiment with take. Type rga and then zg.

Processing

Topspin offers a step-by-step guide to processing. You may follow it by selecting Analysis, T_1/T_2 Relaxation. Or you may simply follow the procedure I've written below.







Processing begins with the fully (or nearly) relaxed spectrum (or slice) in the 2D. Type rser #, where # is the FID that had the longest vd value (if there are 15 tau values, then # may rang from 1 to 15). Type ef and phase this spectrum. After adjusting the phase, save the phasing to both 2D and 1D by clicking on

, , and then to exit .

You should now see a 1D of the fully relaxed spectrum. Next, you will choose which peaks are to be analyzed for T_1 , based on their intensity. Click peak pick  and then add one or more peaks manually by clicking  and select peak with cursor and left click. Click  to export to the





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relaxation analysis routine. Then, click save+return . You also need to define integral regions to be used for determining T1. Click integrate . Click  to define beginning and end of integral; press left button and drag to define. After adjusting integral, export to relaxation module by clicking  and then choosing Export Regions to Relaxation Module. Then, click save+return . Click  to close the 1D spectrum and return to 2D.

Type xf2 to transform all the spectra.

(If at any time you wish to return to the above 1D spectrum, type rsr #.)

Type t1/t2 to enter the relaxation sub-routine. Your points should be automatically picked. To pick them manually, click . To adjust the settings, click . In particular, make sure the function type is set to invrec. If your points are not all being picked, you may also need to increase the number of drift points are then re-pick. Click  to calculate T1. To delete a particular, possibly outlying point, right-click over the point and select delete. If you have picked more than one peak in your spectrum, click + or - to change to the next peak. Click  to generate a report.

Calibration of 180 degree pulse

The inversion-recovery sequence uses a 180 degree pulse. Frequent users may want to calibrate the 180 pulse themselves which may provide more accurate results. Ask for assistance.

Examples of delay lists using 13 values

#	T1 (seconds)			
	0.5	1.0	3.0	10
1	0.001	0.001	0.001	0.01
2	0.0069	0.015	0.041	0.15
3	0.028	0.055	0.16	0.55
4	0.063	0.13	0.37	1.25
5	0.11	0.22	0.66	2.2
6	0.174	0.34	1.04	3.4
7	0.25	0.5	1.5	5.0
8	0.34	0.68	2.04	6.8
9	0.44	0.88	2.66	8.8
10	0.56	1.13	3.38	11.3
11	0.69	1.38	4.2	13.8
12	0.84	1.68	5	16.8
13	2.5	5	15	50