T1 relaxation experiment

Introduction

When an NMR sample sits in the magnet, the applied static magnetic field B_0 will generate the equilibrium magnetization M_0 along +z axis. When a RF pulse is allied to the sample, the net magnetization will be rotated away from +z axis. T1 relaxation (aka longitudinal or spin-lattice) is the process by which the net magnetization goes back to its initial maximum value ($M_{z,eq}$) parallel to B_0 .

The inversion-recovery experiment measures T_1 relaxation times of any nucleus. If the net magnetization is placed along the -z axis, it will gradually return to its equilibrium position along the +z axis at a rate governed by T1. The equation governing this behavior as a function of the time t after its displacement is:

$$M_z(t)=M_{z, ext{eq}}\left(1-2e^{-t/T_1}
ight)$$



The basic pulse sequence consists of an 180° pulse that inverts the magnetization to the -z axis. During the following delay, relaxation along the longitudinal plane takes place. Magnetization comes back to the original equilibrium z-magnetization. A 90° pulse creates transverse magnetization. The experiment is repeated for a series of delay values taken from a variable delay list. A 1D spectrum is obtained for each value of vd and stored in a pseudo 2D dataset. The longer the recycle delay (d1) is, the

more precise the T1 measurement is. Ideally d1 should be set to 5*T1. A rough estimation of the T1 value can be calculated from the null-point value by using T1=tnull/ln2.

Setting up proton T1 Experiment

- 1) To set up a T1 experiment, start with recording a normal proton spectrum to adjust the spectral sweep width **SWH**, acquisition time **aq** and other parameter if necessary.
- Create new dataset and load "Proton_T1" parameter set. Update the parameters with the ones you obtained from last step. The recycle delay D1 should be ~2-5*T1. Adjust NS accordingly to give sufficient S/N (fig 1).
- 3) Edit the "t1delay" by clicking on List at VDLIST line in fig 1. Fig 2 is a good starting list.
- 4) Change the "TD" value for F1 dimension to the number in your VDLIST (fig 3)
- 5) Collect the pseudo 2D T1 dataset

SPECTRUM	PROCPARS ACC	UPARS TITLE PULS	EPROG PE	EAKS II	NTEG	RALS	SAMPLE	STRUCTURE	PLOT	FID	ACQU
∽ѧҥ҄⊎		Probe: 0	CP QCI 6	00S3	H/F	-C/N	I-D-05 2	Z			
General Channel f1	General										
	PULPROG	t1ir			E	Pulse	program fo	r acquisition			
	TD	32786				Time	domain size	е			
	SWH [Hz, ppm]	8196.72	13.6657			Swee	p width				
	AQ [sec]	1.9999460				Acqu	isition time				
	RG	64				Rece	iver gain				
	DW [µsec]	61.000				Dwell	time				
	DE [µsec]	20.00				Pre-s	can-delay				
	D1 [sec]	5.00000000				Relax	ation delay;	1-5 * T1			
	d11 [sec]	0.0299999993				Delay	for disk I/O		[30 ms	ec]	
	DS	0				Numb	per of dumn	ny scans			
	NS	2				Scan	s to execute	e			
	VDLIST	t1delay			E	Variat	ole delay list	:			
	vd [sec]	5.0000000				vd[10)={ 5.0000	000 sec 0.00100	00 sec	}	

Fig 1. ACQUPARS display in "pulse program parameters" view

Fig 2. An example of t1delay list with 8 delays

2 0. 3 0. 4 0. 5 0.	100 250 500			
3 0. 4 0. 5 0.	250			
4 0.	500			
5 0.	200			
C	800			
b 1.	5			
7 3				
8 5				

Fig 3. ACQUPARS display in "all acquisition parameters" view

SPECTRUM PROCPARS ACQUPARS TITLE PULSEPROG PEAKS INTEGRALS SAMPLE STRUCTURE PLOT FID ACQU ∽л s ⊌ झझ₁₂ ≪ с ् Probe: CP QCI 600S3 H/F-C/N-D-05 Z Experiment F2 F1 Frequency axis Width Experiment Receiver k Nucleus t1ir PULPROG E Current pulse program Durations *** Power AQ_mod DQD Acquisition mode Program FnTYPE traditional(planes) nD acquisition mode for 3D etc. ¥ Probe Acquisition mode for 2D, 3D etc. Lists FnMODE QF -NUS 32786 8 Size of fid TD Wobble DS 0 Number of dummy scans Lock Automation 2 NS Number of scans Miscellaneous 1 Loop count for 'td0' TD0 User Routing 0 TDav Average loop counter for nD experiments

Processing

- Process and adjust phase for the dataset. Use rser n (n is the number of total delays) to read out the last fid. Process and phase correct it. On the Adjust Phase toolbar, click Save for spectrum.
- 2) Go back to pseudo 2D T1 dataset by closing the 1D window
- At the command prompt, type xf2 to process only the F2 axis. Type abs2 to baseline correct the rows.
- 4) On the menu bar, click **Applications**.
- 5) On the **Dynamics** button, click the drop-down arrow to see more options and in the list, select **T1/T2 Module**.

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	<u>T</u> 1T2 (t1t2)
	Prepare for Dynamics Center
	Dynamics Center (dync)

6) The flow buttons change to determine the T1 / T2 relaxation times. While executing the steps below, message windows will be displayed. Please read each message thoroughly and follow the instructions. On the Workflow button bar, click Fid

G Back	₩~ <u>F</u> id	Peaks/Ranges	Relaxation	Fitting	>	Calculation	Report
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7) In the Extract a row from 2d data window, click Spectrum



8) Enter Slice Number = **n** (the last one).

e -	—
Spectrum slice must be extracted Fin This Spectrum should correspond to All further data preparation will be do Slice Number =	om the 2d relaxation data. an experiment with the maximum or minimum delay time, one in respect to this spectrum.
	QK <u>C</u> ancel

- 9) On the Workflow button bar, click **Peaks/Rang** A Peaks/Ranges
- 10) In the Define Peaks and/or Integrals window, click Manual Integration.



- 11) Define the regions by drawing an integral over the peaks of interest, On the Integration toolbar, click **Save/export integration regions**
- 12) In the list, select Export Region To Relaxation Module.



13) In the Prepare relaxation data window, click OK



14) On the Workflow button bar, select Relaxation.

Relaxation

- 15) By default, the selected areas are peak-picked, and the first peak is displayed in the Relaxation window.
- 1. . 16) On the Workflow button bar, select **Fitt**ing
- 17) In the message window, click Close.



- 18) In the Relaxation parameters window, click **OK** and select **Area** as Fitting type.
- 19) On the Workflow button bar, select Calculation. > Calculation
- 20) In the message window, click Close.



21) In the T1/T2 tools bar, click Calculate fit for all peaks \bigotimes

Delet Descent
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Region 1 from 7.797 to 7.650 ppm
T1 = 1.339s
Region 2 from 7.313 to 7.159 ppm
T1 = 1.294s
Region 3 from 6.860 to 6.413 ppm
T1 = 555.498m
Region 4 from 4.891 to 4.725 ppm
T1 = 644.916m
Region 5 from 3.414 to 3.321 ppm
T1 = 1.110s
Region 6 from 2.076 to 1.372 ppm
T1 = 398.815m
Region 7 from 1.171 to 0.552 ppm
T1 = 378.896m

🖕 Relaxation parameters							
General Par	ameters						
16	FID # fo	or phase deter	mination				
1000.0	Left lim	it for baseline	correction				
-1000.0	Right li	mit for baseline	correction				
5	Numbe	r of drift points					
1.0E-5	Conver	rgence limit					
16	Numbe	r of points					
1	First sli	ce					
1	Slice in	crement					
1.0 Peak sensitivity							
r Fitting Function							
uxnmrt1							
1		Number of components					
vdlist	•	' List file name					
0.001		Increment (au	ito)				
pd	•	to pick data p	oints				
Iteration con	trol para	imeters					
	Guesse	s	Reset				
Additional P	aramete	rs					
10000.0	(GAMMA(Hz/G)					
10.0	L	LITDEL(msec)					
100.0	E	BIGDEL(msec)					
1.0	(GRADIEN(G/cn	1)				
OK Apply Cancel							

22) On the Workflow button bar, select **Report**

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11	11 -	1.3399			
	10 · · ·	7.976+-85			
1.5					
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14	10.000				
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12	101.000	3.688		4 10051-07	
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22	1.000+	7.688	5.6663+-87	6.457584	
14	1.508+	2.685	7.25184-88	7.535+87	
10	2.668+	1.698	3.4871e+88	4.4145++87	
25	7.508+	7.698	4.2955++88	4.56550-87	
12	3.008+	7.698	4.7555++88	5.5905++87	
10.	1.508+	7.655	5.5423e+88	6.012++07	
28.	4.908+	7.695	5.406e105	6.3695e+62	
10	4.508x	2.698	5.5853e+88	6.3552e+82	
11	5.008+	7.685	5.7016++08	6.7577#+82	
10.					