

KJM 9250

ProtonT1 Spectra on the DRX500 Spectrometer

Version 5.0

Topspin 1.3 Windows XP DRX 500



© Professor Emeritus Alistair Lawrence Wilkins, University of Waikato, New Zealand. April 2018

Proton T₁ Spectra on the DRX-500 Spectrometer

1.0 Introduction

An **awprotont1** parameter set and linked **VDLIST** file have been set up on the DRX500 spectrometer running under **TS1.3**

The **awprotont1** parameter set has D1 = 10 sec and a linked **VDLIST** file with 8 x delays in the range 0.03 to 10 sec with the <u>longest delay (10 sec) the first used VDLIST</u> value and the shortest delay (0.03 sec) the last used value.

D1 and the longest (first) **VDLIST** value should be 3-5 times the longest T_1 in the sample compound.

 T_1 data sets can be processed on the spectrometer terminal or off line using any version of Topspin provided a copy of the linked **VDLIST** file is resident in the experiment's top level folder (= default set up: see below) or a copy of the originally named **VDLIST** file is recreated in the offline terminal's *C:\Bruker\Topspin...\exp\stan\nmr\lists\vd* folder.

uxnmr.info	2/01/2017 10:50 p	INFO File	4 KB
📄 uxnmr.par	2/01/2017 10:50 p	PAR File	24 KB
🗋 vdlist	10/01/2018 8:49 a	File	1 KB

NB: The experimental copy of the variable delay file is named as **vdlist** irrespective of the name of the source vdlist file.

2.0 Experiment Set Up

Create an experiment and read in the awprotont1 parameter set (+ getprosol).
 Pulse programme = t1ir.

Two parameter sets with longer **30 or 60 sec d1 delays (awproton30t1** and **awproton60t1)** and **12 or 16 VDLIST** values respectively have also been created..

- 2) Review default settings. These settings can be adjusted if required. TD(F2) = 32K, TD(F1) = 8 (or 12 or 16 for the 30 or 60 sec VDLIST files respectively) SW = 14 ppm, O1P = 6.3 ppm. D1 = 10 sec. NS = multiple of 2, 4 or 8, DS = 0, 2 or 4.
- 3) Type **ased** (enter) and review other parameters used in the job including the linked **VDLIST** file = **AWPROTONT1-8**
- 4) The VDLIST file should have the entries shown below (next page). Values are in seconds. Do not alter the values in this file. An alternatively named VDLIST file should be created if different VD and D1 values are required for a particular compound

	Search
10s	
6s	
₿s	
1s	
0.5s	
0.25s	
0.1s	
0.00-	

- 5) Set receiver gain using RGA (*important*!).
- Type edp (enter) and check that SI(F2) = 32K, SI(F1) = 8, WDW(F2) = EM, LB (F2) = 0.3-0.5 Hz or other value of your choice.

SI =	32768	8		Size of real spectrum
SF [MHz] =	500.1300000	500.13000	00	Spectrometer frequency
OFFSET [ppm] =	13.301	12.308		Low field limit of spectrum
SR [Hz] =	0.00	0.00		Spectrum reference frequency
HZpPT [Hz] =	0.213709	751.20190	4	Spectral resolution
Window function				
WDW =	EM	🖌 no	~	Window functions for trf, xfb,
LB [Hz] =	0.\$	0.30		Line broadening for em

Experiments with 12 or 16 VDLIST points are processed with SI(FI) = 16.

- 7) Start the acquisition using the **ZG** command.
- 8) When the experiment has run type **rser 1** (enter) to read in the first serial file which will appear in a TEMP screen display window.
 - TEMP 1 1 DuioDRX600-07 alistaiw

 Spectrum
 ProcPars
 AcquPars
 Title
 PulseProg
 Peaks
 Integrals
 Sample
 Structure
 Fid

 Image: Structure
 Fid
 Image: Structure
 Fid
 Image: Structure
 Fid

 Image: Structure
 Fid
 Image: Structure
 Fid
 Image: Structure
 Fid

 Image: Structure
 Fid
 Image: Structure
 Fid
 Image: Structure
 Fid

 Image: Structure
 Fid
 Image: Structure
 Fid
 Image: Structure
 Fid

 Image: Structure
 Fid
 Image: Structure
 Fid
 Image: Structure
 Fid

 Image: Structure
 Fid
 Image: Structure
 Fid
 Image: Structure
 Fid

 Image: Structure
 Fid
 Image: Structure
 Fid
 Image: Structure
 Fid

 Image: Structure
 Fid
 Image: Structure
 Fid
 Image: Structure
 Fid

 Image: Structure
 Fid
 Image: Structure
 Fid
 Image: Structure
 Fid

 Image: Structure
 Fid
 Image: Structure
 Fid
 Image: Structure
 Fid
- 9) Type EFP (return) to transform it and phase it as per a normal proton spectrum.

10) Type **edp** (enter) and note the phase constants for this spectrum.

Phase correction		2
PHC0 [degree] =	185.998	
PHC1 [degree] =	0.019	
PH_mod =	pk	~

- 11) Close the **TEMP** window and reload the T_1 data set file.
- 12) Type edp (enter) and enter the phase constants noted in step 10 above into the F2 PHC0 and PHC1 cells and check PH_MOD = pk. F1 cell info is not used.

Phase correction			1999 1999		
PHC0 [degree] =	185.998		0.000		Oth order correction for pk
PHC1 [degree] =	0.019		0.000		1st order correction for pk
PH_mod =	pk	~	mc	*	Phasing modes for trf, xfb,

13) Type **xf2** (return) to transform the 2D data set followed by **abs2** (return) to baseline smooth it.

14) Provided phase constants have been correctly set up the transformed data set plot should resemble that shown below. Black = a positively phased signal, red = a negatively phased signal.



3.0 T₁ Data Set Processing

15) Open Topspin's Analysis menu and click its T1/T2 Relaxation tab.



This will open up the screen display shown below.



16) Click the **Extract Slice** button and then the click the **Spectrum** button in the panel that appears.



17) Enter 1 in the Slice Number cell and click the OK button. This will display the transformed spectrum ex the first (longest) VDLIST value = the one that was phased via the rser 1 routine in steps 8 and 9.

43		
This Spectrum shou	t be extracted From the 2d rela ald correspond to an experime	ixation data. It with the maximum or minimum delay time
All further data prep	paration will be done in respect	to this spectrum.
All further data prep	Slice Number =	to this spectrum.



18) Close the screen message that appears and click the **Define Ranges** button.

19) Expand the spectrum that appears in the usual way and integrate selected peaks in it. Integrals should start and terminate as close as possible to the edge of a peak.

E - row Mous 7.78	l from "50 se Sensitiv 31 ppm / 38	0 T1-T2" ity: 1.0 91.50 Hz	211 1	D:\uio\DR	x500-07 a	listaiv	 (
₽ DEFI Defi Retu	INE REGION .ne: Drag u urn: Left-c	MODE sing left lick high	mouse bu lighted i	itton .con			
							
4							
₽ 	m				4.6		
•	0000				0.9708 1.9708		 .0743

20) Click the Floppy Disk icon button (7th from the left hand side of the menu bar in TS1.3) and select its **Export Regions to Relaxation Module and .ret**. tab.



21) Click the **Relaxation Window** button, note any screen messages that may appear and close them. Select **Intensity** or **Area** in the plot window that appears. **Area** is invariably the better choice.



22) Click the Fitting Function button and note the comments about intensity or area options in the screen display that appears and close it.

unction			٤
R			
ease select the functi	ion to which the pe	ak intensities or integ	grals
e to be fitted, depend	ling on the experin	nent which produced	the relaxation data.
stillings dialog provide:	s all possibilities io	Relaxation analysis	aujustment.
			Close

23) When the Fitting Function message screen is closed a panel with relaxation parameters (split into two screen will appear.

	anamotoro	and a second second and			
1	FID # for	phase determination			
1000.0	Left limit	for baseline correction			
-1000.0	Right lim	it for baseline correction			
5	Number	of drift points			
1.0E-5	Converg	ience limit			
8	Number	of points			
1	First slic	e			
4	Slice inc	rement	Iteration contr	ol parameters Guesses	Reset
1			C		<i></i>
Fitting Fur	nction	رك. ۱۰ ۱	Additional Par	rameters	
Fitting Fur	nction	Function Type	Additional Par 10000.0	rameters GAMMA(Hz/G)	
Fitting Fur uxnmrt1	nction -	Function Type Number of components	Additional Par 10000.0 10.0	GAMMA(Hz/G)	
Fitting Fur uxnmrt1 1 vdlist	retion •	Function Type Number of components List file name	Additional Par 10000.0 10.0 100.0	GAMMA(Hz/G) LITDEL(msec) BIGDEL(msec)	
Fitting Fur uxnmrt1 1 vdlist 0.0010	retion •	Function Type Number of components List file name Increment (auto)	Additional Par 10000.0 10.0 100.0 1.0	GAMMA(Hz/G) LITDEL(msec) BIGDEL(msec) GRADIEN(G/cr	n)

- **24**) Check **Function Type = uxnmrt1** and **List file name = vdlist**. Other cells/values can be left as they are (= default settings)
- 25) Click the double red arrow in the menu bar at the <u>top left</u> of the plot window and <u>NOT</u> the single red arrow button in the NMR Relaxation Guide menu below the Fitting Function button.



- **26)** The + and buttons in the upper menu bar can be used to advance (or reverse) the individual T_1 plots.
- 27) Fitting type (Intensity or Area) can be changed in the plot display. If this is done clicking the **double red arrow** will recalculate the T₁ results and update their plots.
- 27) The NMR Relaxation Guide has buttons which can be used to display and/or print T_1 results. A sample report for one signal is shown below.

```
Dataset :
D:/uio/DRX500-07/data/alistaiw/nmr/500 T1-T2/211/pdata/1
AREA fit :
I[t]=I[0]+P*exp(-t/T1)
8 points for Integral 1, Integral Region from 8.723 to 8.660 ppm
Results
           Comp. 1
I[0]
         1.060e+000
        -2.000e+000
т1
     =
            2.998s
SD
         1.068e-002
                integral intensity
   tau ppm
            8.681 9.9096e+008 2.1672e+007
  10.000s
   6.000s
             8.681 7.6799e+008 1.6613e+007
              8.681 3.1511e+008 6.7054e+006
   3.000s
              8.681 -3.5332e+008 -7.9351e+006
   1.000s
 500.000m
              8.681 -6.1785e+008 -1.3552e+007
 250.000m
             8.681 -7.7128e+008 -1.6694e+007
             8.681 -8.7338e+008 -1.9022e+007
 100.000m
  30.000m
             8.681 -9.2286e+008 -2.0054e+007
```