

KJM 9250

ProtonT1 Spectra on the AVIII800HD Spectrometer

Version 1.0

Topspin 3.5 Windows 7 AVIII HD 800



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ProtonT₁Spectra on the AVIII800HD

1.0 Introduction

Three **aw coded protont1** parameter sets and linked **VDLIST files** have been set up on the **AVIIIHD-800** spectrometer running under **TopSpin3.5**.

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.

The **awproton30t1** and **awproton60t1** parameter sets have **D1** and **VD**_{max} = 30 or 60 sec respectively and linked **VDLIST** files with **12** or **16** points respectively.

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

1) Create an experiment and use the **rpar** command to read one of the following parameter sets:

```
awprotont1 (d1 = 10 sec) + getprosol,
or: awproton30t1 (d1 = 30 sec) + getprosol,
or: awproton60t1 (d1 = 60 sec) + getprosol.
Pulse programme = t1ir.
```

- 2) Review default settings. These settings can be adjusted if required. TD(F2) = 64K or 32K. TD(F1) = 8 (or 12 or 16 for the 30 or 60 sec expts respectively: see point 4). SW = 14 ppm, O1P = 6.3 ppm. D1 = 10 sec (or 30 or 60 sec). NS = 1 or a 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 AWPROTONT1-8 VDLIST file should have the entries shown below. 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.

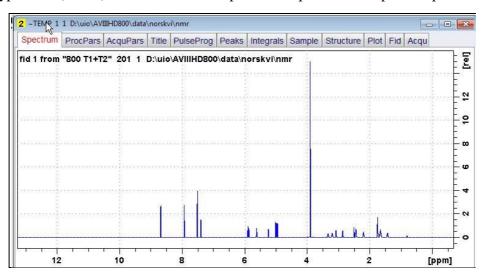
	le Edit ၖearch
1	10s
2	6s
3	3s
4	1s
5	0.5s
6	0.25s
7	0.1s
8	0.03s

Additional **VD** values are present in the **VDLIST** files linked to the **awproton30t1** and **awproton60t1** parameter sets. These files have **12** and **16** points respectively.

- 5) Set receiver gain using RGA(important!).
- 6) Type edp (enter) and check that SI(F2) = 32 or 64K, SI(F1) = 8, WDW(F2) = EM, LB (F2) = 0.3-0.5 Hz or other value of your choice. Data sets with 12 or 16 VDLIST points are processed with SI(F1) = 16.

	F2	F1	Frequency axis
Reference			
SI	65536	8	Size of real spectrum
SF [MHz]	800.0300000	800.0300000	Spectrometer frequency
OFFSET [ppm]	13.27518	9.70782	Low field limit of spectrur
SR [Hz]	0	0	Spectrum reference freq
HZpPT [Hz]	0.170299	1.251948	Spectral resolution
SPECTYP	UNDEFINED		▼ Type of spectrum e.g. C
Mindow func	tion		
WDW	EM	▼ SINE	 Window functions for trf,
LB [Hz]	0.30	0.30	Line broadening for em

- 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.
- 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.

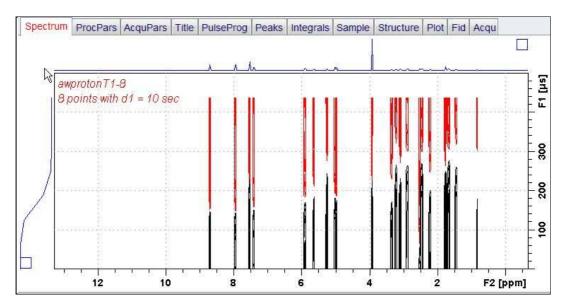
Phase correct	tion
PHC0 [degrees]	27.243
PHC1 [degrees]	-98.137
PH_mod	pk 🔫

- 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.

🔊 Pisase correc	tion		
PHC0 [degrees]	27.243	0	Oth order correction for p
PHC1 [degrees]	-98.137	0	1st order correction for p
PH_mod	pk	▼ mc	 Phasing modes for trf, xf

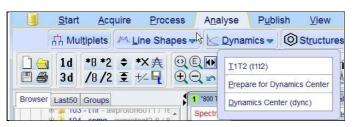
13) Type **xf2** (return) to transform the 2D data set followed by **abs2** (return) to baseline smooth it.

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

 Click Topspin's Analyse menu tab then its Dynamics sub-menu tab and click its T1T2 tab.



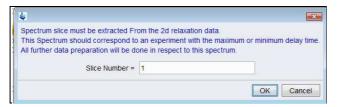
16) This will open the T1/T2 processing menu bar shown below.



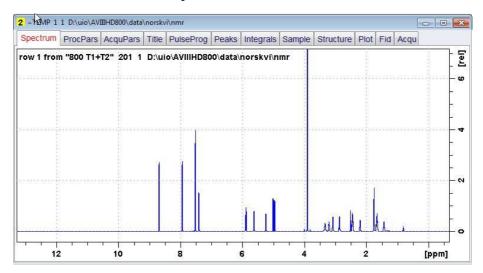
Click the **FID** tab and click the **Spectrum** button in the screen panel that opens.

2	Fid or Spectrum must be extracted From the 2d relaxation data.
	This row should correspond to an experiment with the maximum or minimum delay time. All further data preparation will be done in respect to this row.

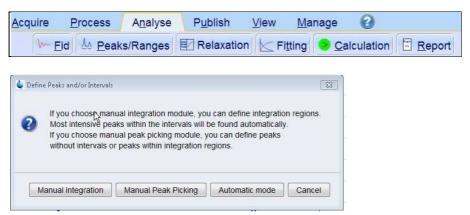
17) The screen panel shown below will appear.



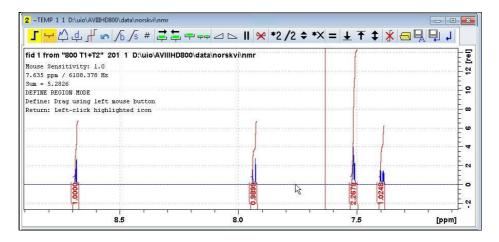
Enter 1 in the **Slice Number** cell and click the **OK** button. This will display the transformed spectrum ex the first (longest) **VDLIST** value = the one that was phased via the **rser 1** routine in steps 8 and 9.



18) Expand the spectrum that appears in the usual way and click the **Peaks/Ranges** menu bar tab.



Select the **Manual integration** tab, close any screen messages that appear, and integrate selected peaks in the usual way.



19) Click the "**Save Region As**" icon menu bar button (= *the floppy disk icon with A below it*) and then its **Export Regions to Relaxation Module and .ret**. tab.

< = ± Ŧ \$)	
	Save Regions To 'intrng' Save Regions To 'reg'
	Export integration regions
	Export Regions To Relaxation Module and .ret. Save & Show List

20) Click the **Relaxation** menu tab 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 (next page).

<u>A</u> cquire <u>F</u>	Process	A <u>n</u> alyse	P <u>u</u> blish	<u>V</u> iew	<u>M</u> anage	0	
<u>₩ </u> <u>E</u> id	A Peak	s/Ranges	Relaxation	Fittir	ng 😕 <u>C</u> alc	ulation	Report

(plot window: \rightarrow next page)

<mark>> > asc</mark> — +	🕈 📰 🛄 19 89 19 🔄 🖱 🚺 🚹	
Fitting type Intensity Area	T1 I[I]=I[0]+P*exp(-t/T1) Region 1 from 8.700 to 8.674 ppm	
l of 4	6.79 s	E
Data preparation is o	or	
	0	
	0 0	

21) Click the Fitting menu bar tab and note the comments about intensity or area options in the screen display that appears and close it

<u>A</u> cquire	<u>P</u> rocess	A <u>n</u> alyse	P <u>u</u> blish	<u>V</u> iew	<u>M</u> anage	0	
Im E	id 🌆 <u>P</u> eak	(s/Ranges	Relaxation	n K Fit	tting 😕 <u>C</u>	alculation	Report
	maa nika		а а	12	.)		
🖕 Fitting Fund	tion 🗟				23		
	se select the funct		beak intensities or ir iment which produc		tion data		
			for Relaxation analy				
4				1	Close		

22) When the **Fitting** function message screen is closed a panel with **Relaxation parameters** (split into two screen captures below) will appear.

	arameters		
1	FID # for phase determination		
1000.0	Left limit for baseline correction		
-1000.0	Right limit for baseline correction		
5	Number of drift points		
1.0E-5	Convergence limit		
8	Number of points		2
1	First slice	Iteration contr	ol parameters Guesses Reset
1	Slice increment		Л
57.5355 (1966)		-Additional Par	ameters
Fitting Fur	nction	10000.0	GAMMA(Hz/G)
r nung r u			LITDEL(msec)
53	✓ Function Type	10.0	LIIDEL(IIISEC)
53	Function Type Number of components	10.0	BIGDEL(msec)
uxnmrt1 1			
uxnmrt1	Number of components	100.0	BIGDEL(msec)

- **23**) Check that **Function Type = uxnmrt1** and **List file name = vdlist**.
- **24**) Click the **double red arrow** in the T1 plot's menu bar and <u>NOT</u> the **Calculation** tab in the upper menu bar.

*8 *2 \$ @					
	2 Relaxation "800 T1+T2" 20	21 1 D\uio\AVIIHD800\data\norskvi\nmr			
5 - awcpmgpg - 1 7 - t1irpg - 13C T 3 - awcpmgpg - 1 1 - t1ir - awproto 1 - cpmg - awpro	Fitting type Intensity Area 4 of 4	T1 [[]]=![0]+P*exp(4/T1) Region 4 from 7.408 to 7.384 ppm []] T1=2.98 []]			
7 - t1irpg - 13C T 3 - awcpmgpg - 1) - awcpmgpg - 1 1 - awcpmgpg - 1 2 - t1irpg - 13C T 3 - awcpmgpg - 1	Region 1 from 8.700 to T1 = 3.177s Region 2 from 7.949 to T1 = 3.341s Region 3 from 7.529 to				
	T1 = 1.154s Region 4 from 7.408 to T1 = 2.900s	₽			
	۲ m •	0 2 4 6 8 10[s]			

- 25) The + and buttons in the upper menu bar can be used to advance (or reverse) the individual T_1 plots.
- **26**) 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 **Report** tab in the .menu bar can be used to view fitting report. A sample report for one signal is shown below.

cquire	<u>P</u> rocess A <u>n</u> alyse P <u>u</u> blish <u>V</u> iew <u>M</u> anage 🚱
W~	<u>Fid</u> 🚣 <u>P</u> eaks/Ranges 🖾 Relaxation 🖂 Fitting 📀 <u>C</u> alculation 🖾 <u>R</u> eport
	tting report
Service (113)	Edit Search
File	
1	pataset :
2	D:\uio\AVIIIHD800\data\norskvi\nmr/800 T1+T2/201/pdata/1
3	AREA fit :
4	I[t]=I[0]+P*exp(-t/T1)
6	8 points for Integral 4, Integral Region from 7.410 to 7.378 ppm
7	Results Comp. 1
8	Kesuits comp. 1
9	I[0] = 1.062e+000
10	P = -2.043e+000
11	T1 = 2.897s
12	SD = 3.402e-003
13	
14	tau ppm integral intensity
15	
16	10.000s 7.401 1.7097e+009 5.2e+007
17	6.000s 7.401 1.3686e+009 4.1392e+007
18	3.000s 7.401 5.6961e+008 1.6667e+007
19	1.000s 7.401 -6.5005e+008 -2.0877e+007
20 21	500.000m 7.401 -1.1172e+009 -3.5464e+007
21	250.000m 7.401 -1.3877e+009 -4.3569e+007 100.000m 7.401 -1.5619e+009 -4.9122e+007
22	100.000m 7.401 -1.5619e+009 -4.9122e+007 30.000m 7.401 -1.649e+009 -5.1739e+007
24	30.000m /.401 -1.04907009 -3.1/390400/