KJM 9250

Proton T₂ Spectra on the DRX500 Spectrometer

Version 5.0

Topspin 1.3 Windows XP DRX 500

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1.0 Introduction

An **awprotont2** parameter set and linked **VCLIST** file from which a **VDLIST** file _must_ be calculated prior to processing a T$_2$ data set have been set up on the DXR-500 spectrometer running under TS1.3.

The parameter sets have **D1 = 10 sec** and 8 x **VCLIST** values. The T$_2$ data set is acquired using the _smallest vclist value first. Prior to processing T$_2$ data sets variable constant (VC) values must be converted to time domain variable delay (VD) values using the vctovd command (see instructions 15 and 16.)_

D1 and the longest **VD** time derived from the largest VC **value** should be 3-5 times the longest T$_2$ in the sample compound. T$_2$ is always shorter than T$_1$.

$$VD_{max} = [P2 + (2*D20)]* VC_{max}$$

T$_2$ data sets can be processed on the spectrometer terminal or off line using any version of Topspin provided the linked **VCLIST** file is either resident in the experiment's top level folder (= default setting: see below) or recreated or copied into the off line terminal's _C:\Bruker\TopspinX.X\exp\stan\nmr\lists\vc_ folder.

If required the VCLIST file that is present in the experiment's top level folder can be opened with **WordPad** and the **VC** values in it can be viewed and used to recreate the original named **VCLIST** file from which the **VDLIST** file can be created using the **vctovd** command.

uxnmr.info	2/01/2017 10:50 p....	INFO File	4 KB
uxnmr.par	2/01/2017 10:50 p....	PAR File	24 KB
vclist	8/02/2018 10:30 p....	File	1 KB

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

2.0 Experiment Set Up

1) Create an experiment and read in the **awprotont2** parameter set **(+ getprosol).**
Pulse programme = **cpmg**

2) Review default settings. These settings can be adjusted if required.
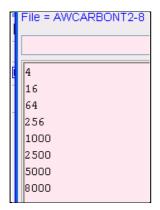**TD(F2) = 32K, TD(F1) = 8**
SW = 14 ppm, O1P = 6.3 ppm.
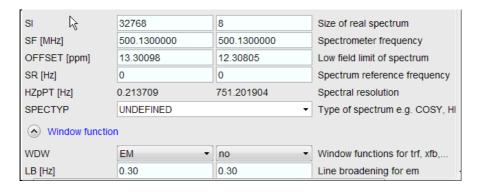**D1 = 10 sec.**
D20 = 600 usec = 0.000600 sec
NS = multiple of 2, 4 or 8, DS = 0, 2 or 4.

3) Type **ased** (enter) and review other parameters used in the job including the linked
VCLIST file = **AWPROTONT2-8.**

4) The **VCLIST** file should have the entries shown below, or other similar values. Do
not alter the values in this file. An alternatively named **VCLIST** file should be
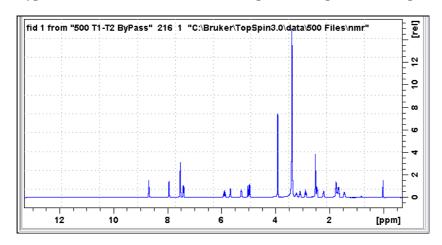created if different **VC** and **D1** values are required for a particular compound.

```
File = AWCARBONT2-8

4
16
64
256
1000
2500
5000
8000
```

5) Set receiver gain using **RGA** *(important!).*

6) 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	500.1300000	Spectrometer frequency
OFFSET [ppm]		13.30098	12.30805	Low field limit of spectrum
SR [Hz]		0	0	Spectrum reference frequency
HZpPT [Hz]		0.213709	751.201904	Spectral resolution
SPECTYP		UNDEFINED		Type of spectrum e.g. COSY, HI
Window function				
WDW		EM	no	Window functions for trf, xfb,...
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
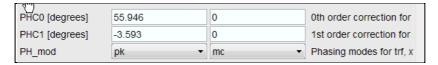will appear in a TEMP screen display window.

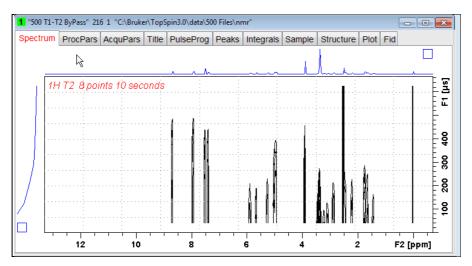**9)** Type **EFP** (return) to transform it and phase it as per a normal proton spectrum.

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

PHC0 [degrees]	55.946
PHC1 [degrees]	-3.593
PH_mod	pk

11) Close the **TEMP** window and reload the T_2 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.
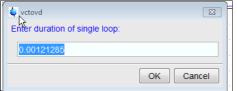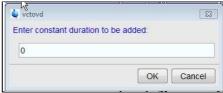
PHC0 [degrees]	55.946	0	0th order correction for
PHC1 [degrees]	-3.593	0	1st order correction for
PH_mod	pk	mc	Phasing modes for trf, x

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. Signals intensities go from their maximum positive value to zero intensity.

3.0 T₂ Data Set Processing

15) _Prior to processing a T₂ data set it is essential that variable constant (VC) values are converted to time domain variable delay (VD) values._

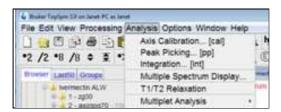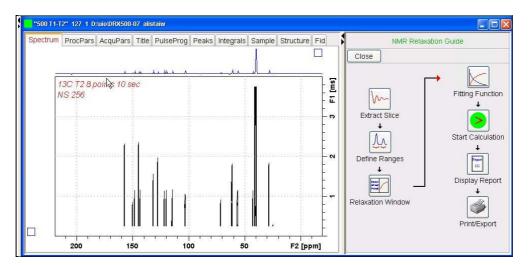**16)** Type **vctovd** (enter) and click OK on the display panels that appear.

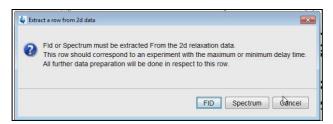See the Appendix for the calculation of this value.   Not used.   .

17) Open Topspin's **Analysis** menu and click its **T1/T2 Relaxation** tab. If other Bruker processing software has been installed on the spectrometer terminal, as may be the case on the AVII-600, select the **Analysis** menu's **Topspin T1/T2 module** tab and open its **T1/T2 Relaxation** sub-menu tab.
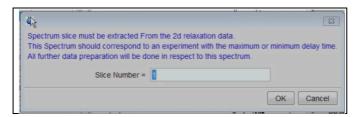

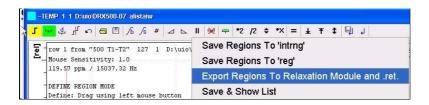This will open up the screen display shown below.

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18) Click the **Extract Slice** button and then the click **Spectrum** button in the panel that appears.
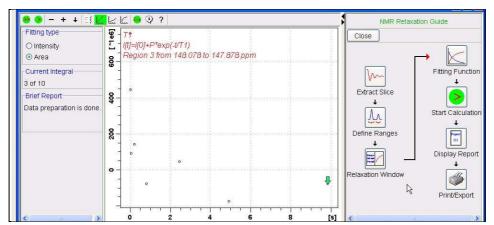
19) Enter **1** in the **Slice Number** cell and click the **OK** button. This will display the transformed spectrum ex the first (shortest) **VCLIST → VDLIST** value = the one that was phased via the **rser 1** routine in steps 8 and 9.

20) 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.

21) Click the "**Save Region As**" icon button (= 7th from the left in the upper menu bar: *the one with the floppy disk icon*) and then its **Export Regions to Relaxation Module and .ret**. tab.

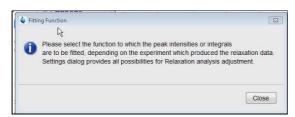**22)** Click the **Relaxation Window** button and select **Intensity** or **Area** in the plot window that appears. **Area** is usually the better choice.
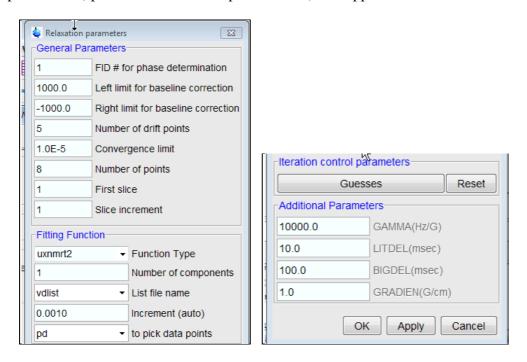

The appearance of T1 information in this screen is a long standing Topspin bug.
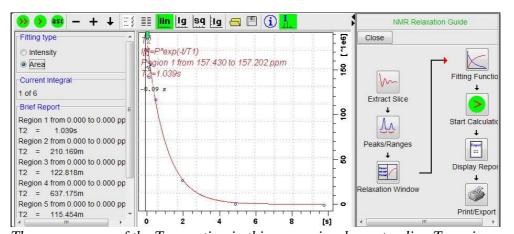
23) Click the **Fitting Function** button and note the comments about **intensity** or **are**a options in the screen display that appears and close it.

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

25) Check **Function Type = uxnmrt2** *(important!)* and **List file name = vdlist**.
Other cells/values can be left as they are (= default settings).
Click **Apply** and **OK** and close the window.

26) Click the **double red arrow** in the menu bar at the top left of the plot window and **NOT** the single red Start Calculation arrow button in the NMR Relaxation Guide menu below the Fitting Function button.

The appearance of the T_1 equation in this screen is a long standing Topspin error.

27) The + and - buttons in the upper menu bar can be used move through the series of T_2 plots.

28) Fitting type (**Intensity** or **Area**) can be changed in the plot display. If this is done clicking the **double red arrow** will recalculate the T_2 results and update their plots.

29) The NMR Relaxation Guide has buttons which can be used to display and/or print T_2 results. A sample report for one signal is shown below. Zero value points (= completely T_2 relaxed) will be eliminated.

```
Fitting report                                                    [23]
File  Edit  Search
 1  Dataset :
 2    C:\Bruker\TopSpin3.0\data\500 Files\nmr/500 T1-T2 ByPass/216/pdata/1
 3  AREA fit :
 4    I[t]= P*exp(-t/T2)
 5
 6  8 points for Integral 1,  Integral Region from 0.000 to 0.000 ppm
 7  Results      Comp. 1
 8
 9  P    =    9.147e-001
10  T2   =        1.994s
11  SD   =    4.210e-002
12
13      tau     ppm      integral     intensity
14
15     4.851m    8.680  1.1457e+009  2.7347e+007
16    19.406m    8.680  1.0785e+009  2.6697e+007
17    77.622m    8.680  9.7667e+008  2.3493e+007
18   310.490m    8.680  8.4007e+008  1.9537e+007
19     1.213s    8.680  5.5076e+008  1.2981e+007
20     3.639s    8.679  2.1239e+008  5.0751e+006
21     7.277s    8.679  4.7105e+007  1.1544e+006
22    14.554s    8.680  7.0986e+006  2.5912e+005
```