KJM 9250

ProtonT_1 Spectra on the DRX500 Spectrometer

Version 5.0

Topspin 1.3 Windows XP DRX 500

University of Waikato, New Zealand.
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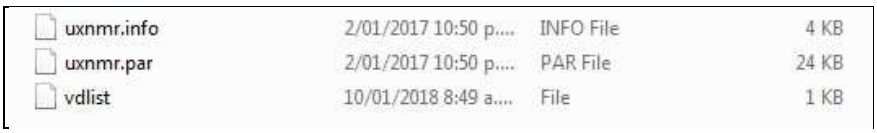
1.0 Introduction

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 *longest delay (10 sec) the first used VDLIST 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.



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

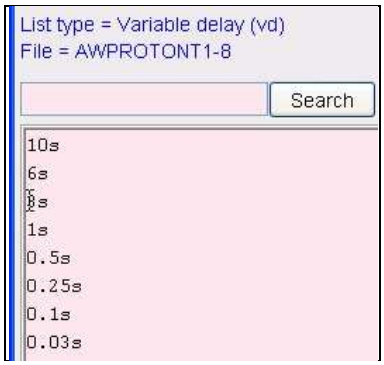
1) Create an experiment and read in the **awprotont1** parameter set **(+ getprosol).** Pulse programme = **t1ir**.

   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.**

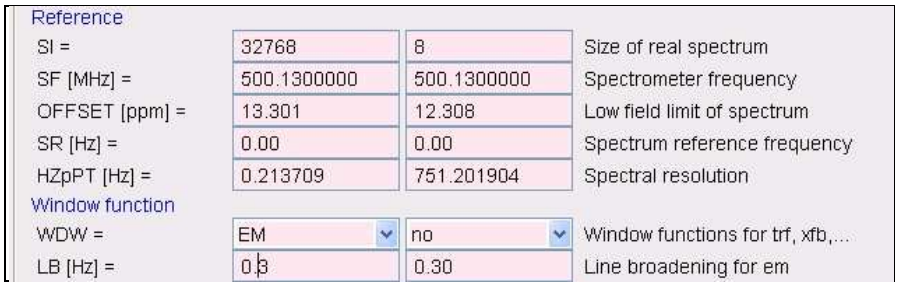
3) Type **ased** (enter) and review other parameters used in the job including the linked **VDLIST** file = **AWPROTONT1-8**

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



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.

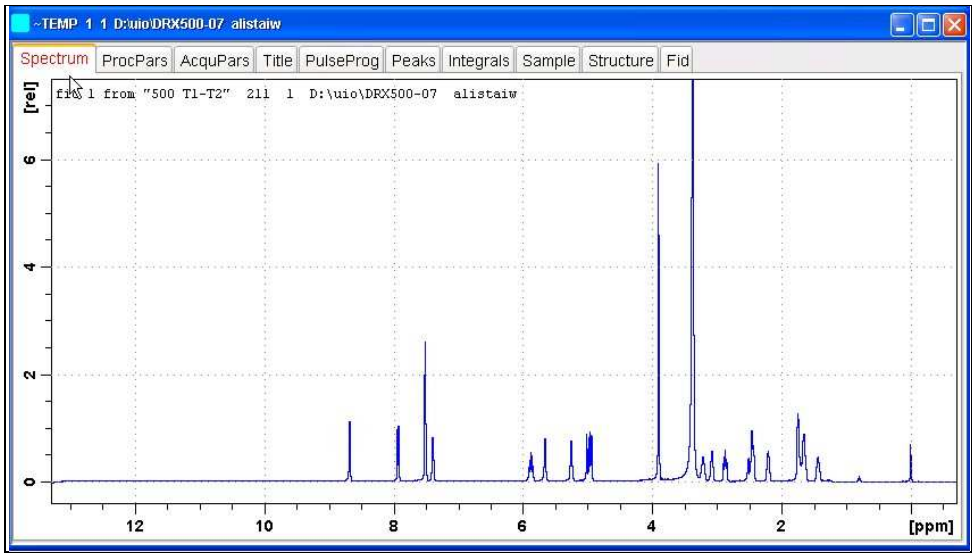


*Experiments with **12 or 16 VDLIST** points are processed with **SI(FI) = 16.***

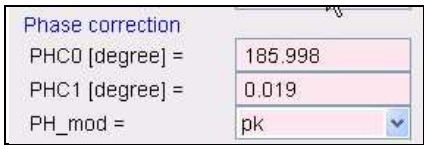
7) Start the acquisition using the **ZG** command.

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.

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

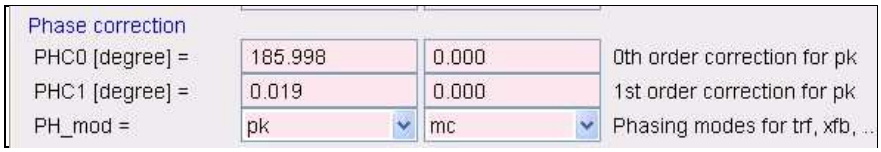


10) Type **edp** (enter) and note the phase constants for this spectrum.



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.



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.

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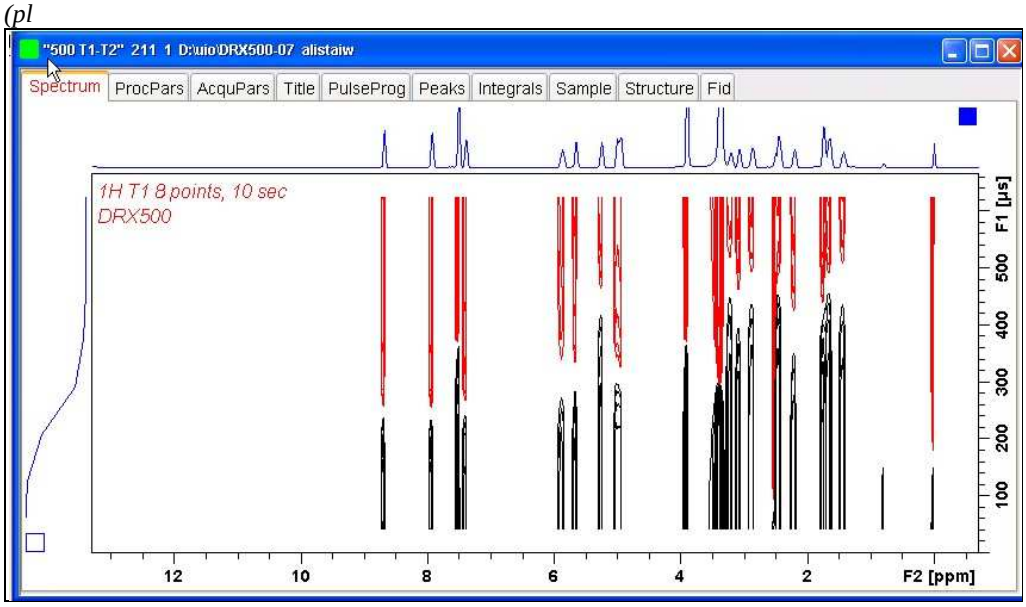
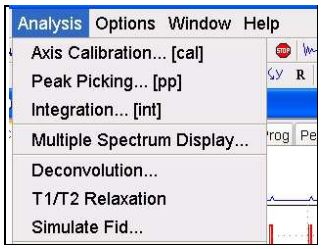
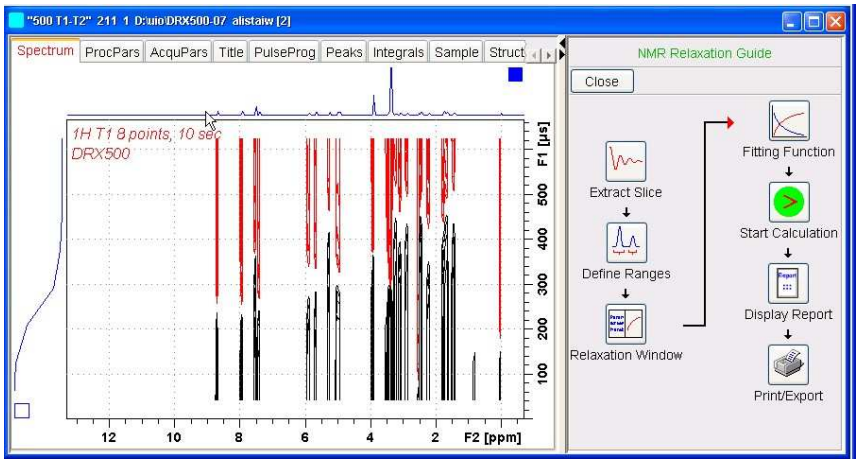


3.0 T_1 Data Set Processing

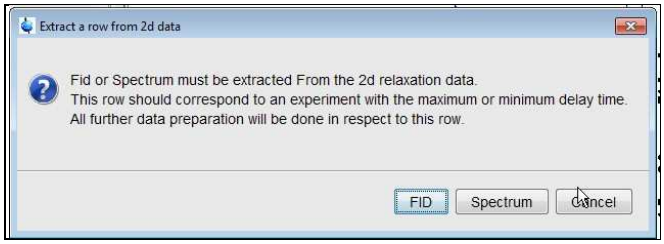
15) Open Topspin's **Analysis** menu and click its **T1/T2 Relaxation** tab.



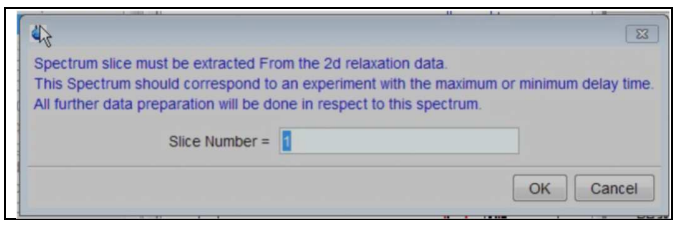
This will open up the screen display shown below.



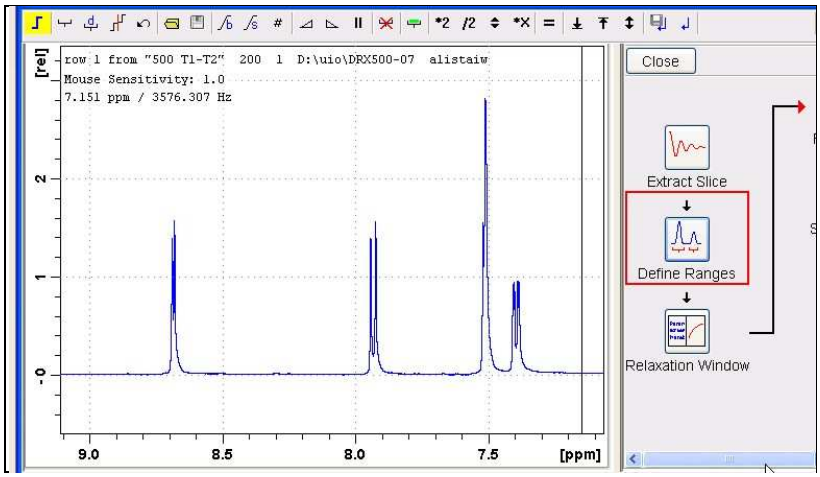
16) Click the **Extract Slice** button and then the click the **Spectrum** button in the panel that appears.



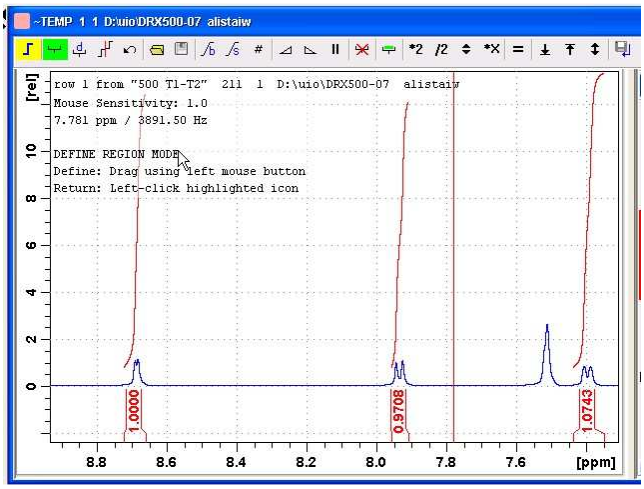
17) Enter **1** in the **Slice Number** cell and click the **OK** button. This will display the transformed spectrum ex the first (longest) **VDLIST** value = the one that was phased via the **rser 1** routine in steps 8 and 9.



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

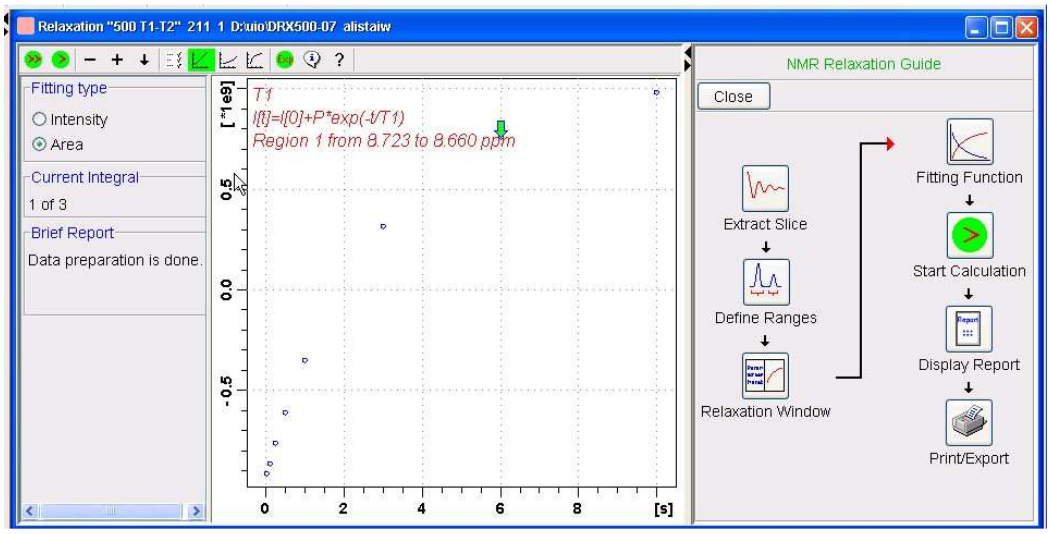


19) Expand the spectrum that appears in the usual way and integrate selected peaks in it. Integrals should start and terminate as close as possible to the edge of a peak.

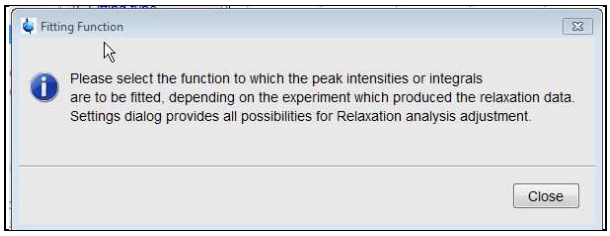


20) Click the Floppy Disk icon button (7th from the left hand side of the menu bar in TS1.3) and select its **Export Regions to Relaxation Module and .ret**. tab.

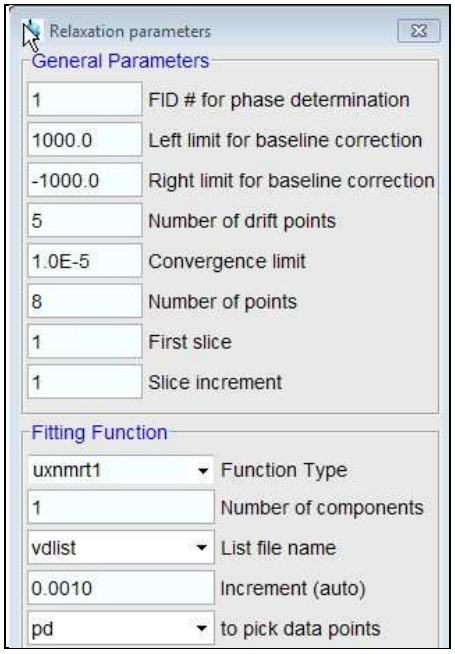
21) Click the **Relaxation Window** button, note any screen messages that may appear and close them. Select **Intensity** or **Area** in the plot window that appears. **Area** is invariably the better choice.

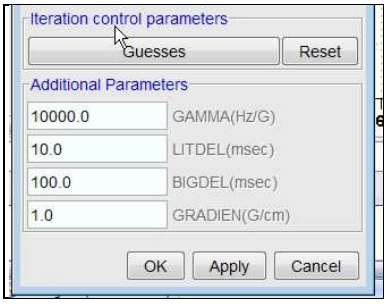


22) Click the **Fitting Function** button and note the comments about **intensity** or **are**a options in the screen display that appears and close it.



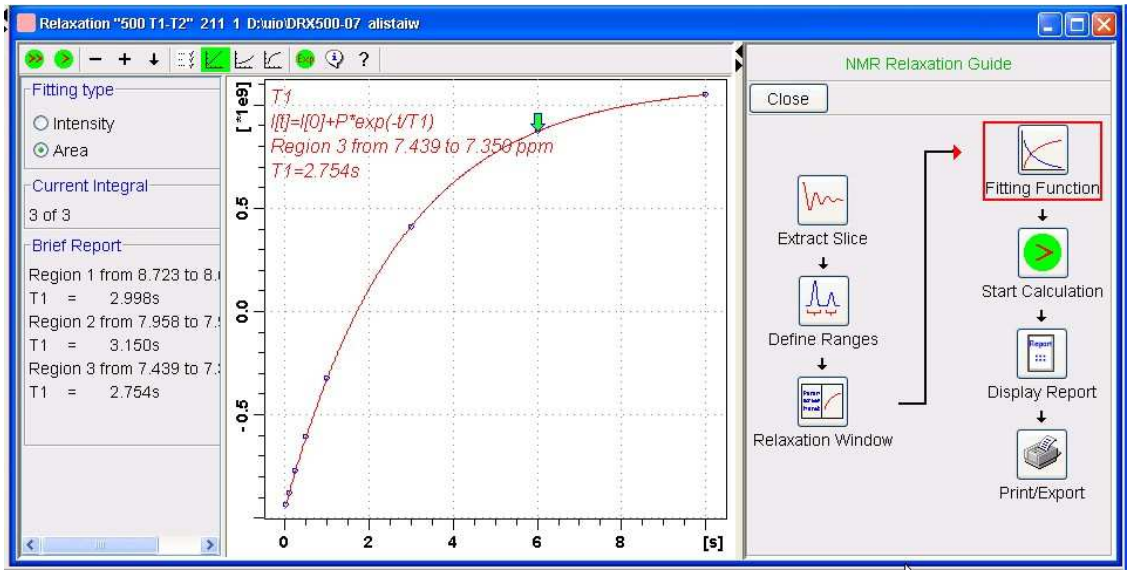
23) When the Fitting Function message screen is closed a panel with relaxation parameters (split into two screen will appear.





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 *top left* of the plot window and **NOT** 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 T_1 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
P     =   -2.000e+000
T1    =       2.998s
SD    =   1.068e-002

    tau    ppm     integral    intensity

   10.000s     8.681  9.9096e+008  2.1672e+007
    6.000s     8.681  7.6799e+008  1.6613e+007
    3.000s     8.681  3.1511e+008  6.7054e+006
    1.000s     8.681 -3.5332e+008 -7.9351e+006
  500.000m     8.681 -6.1785e+008 -1.3552e+007
  250.000m     8.681 -7.7128e+008 -1.6694e+007
  100.000m     8.681 -8.7338e+008 -1.9022e+007
   30.000m     8.681 -9.2286e+008 -2.0054e+007
```