KJM 9250

Proton T_1 Spectra on the AVI-600 and AVII-600

Version 1.0

University of Waikato, New Zealand.
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1.0 Introduction

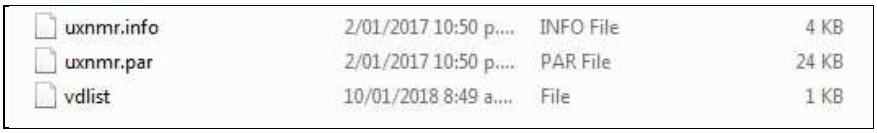
An **awprotont1** parameter set and linked **VDLIST** file have been set up on the AVI-600 and AVII-600 spectrometers running under TS2.1 and TS3.2 respectively.

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.

The **awproton16t1** parameter set with **VD_{max}** and **D1 = 16 sec** can be used for signals with longer T_1's

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.

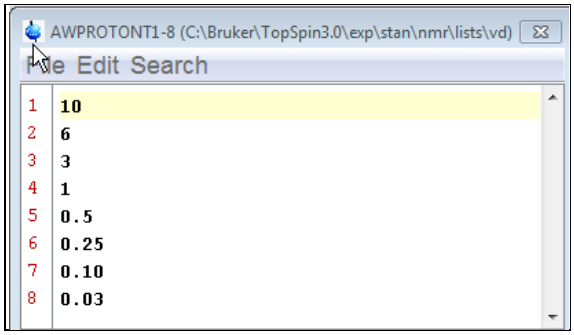
2.0 Experiment Set Up

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

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.
NS = **multiple of 4 or 8, DS = 4 or 8.**

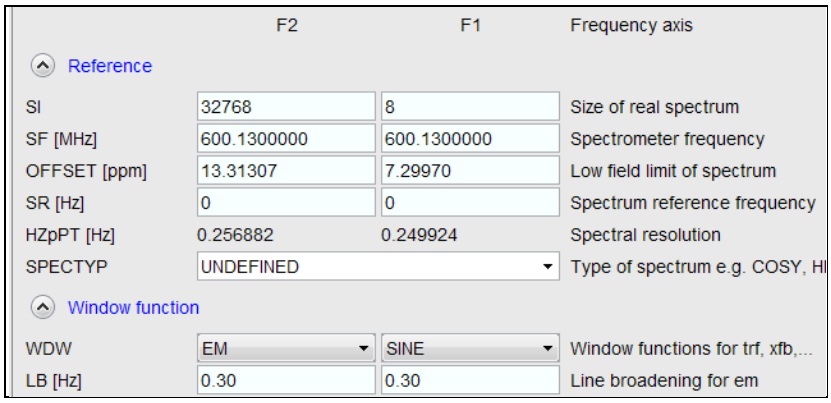
3) Type **ased** (enter) and review other parameters used in the job including the linked **VDLIST** file = **AWPROTONT1-8**

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!).*

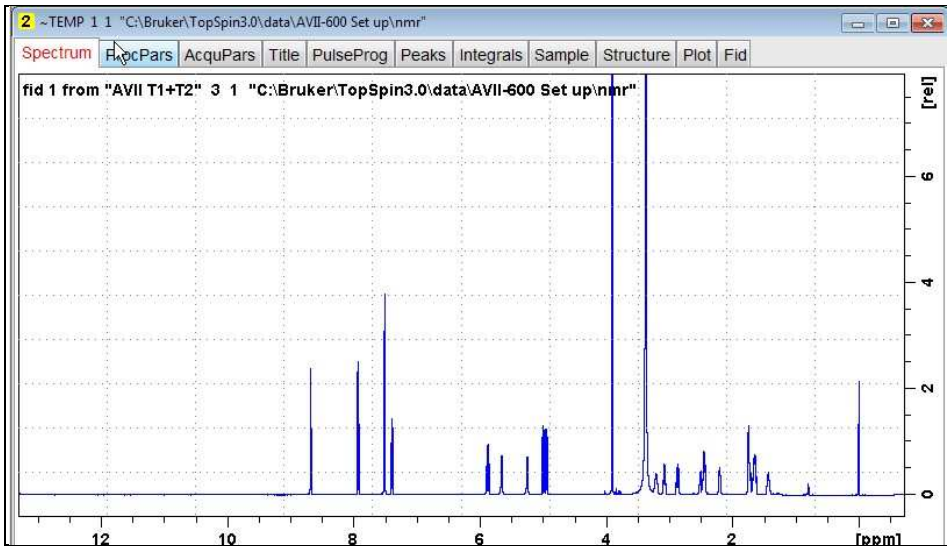
6) Type **edp** (enter) and check that **SI(F2) = 32K**, **SI(F1) = 8, WDW(F2) = EM, LB (F2) = 0.3-0.5 Hz** or other value of your choice.



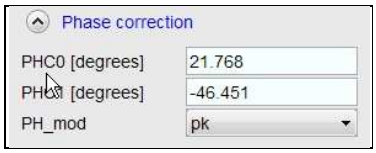
7) Start the acquisition using the **ZG** command.

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.

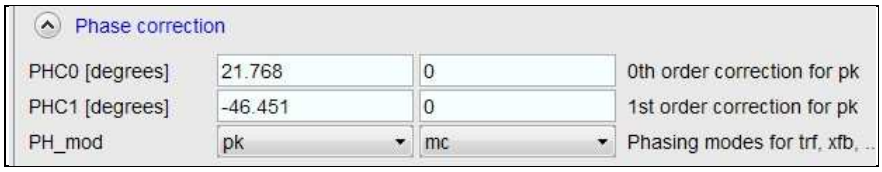


10) Type **edp** (enter) and note the phase constants for this spectrum.



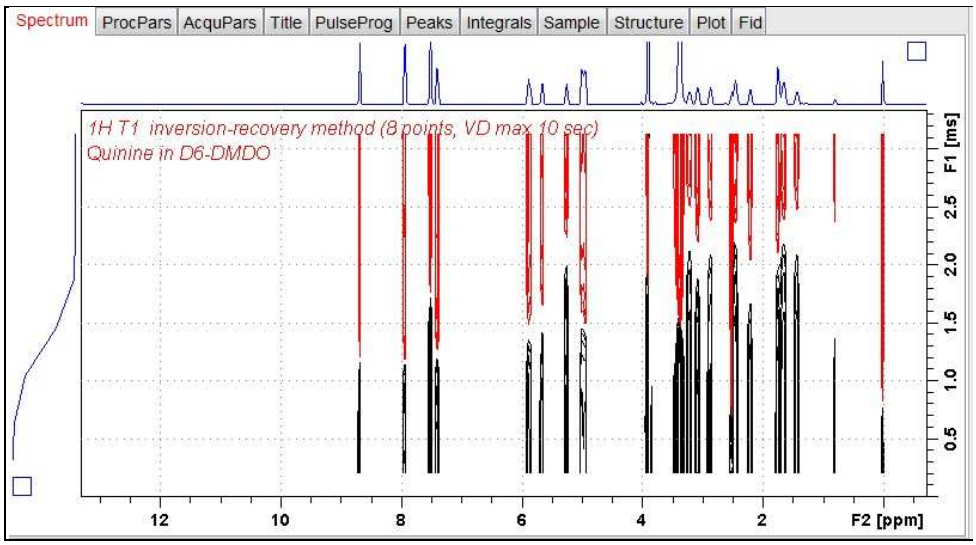
11) Close the **TEMP** window and reload the T_1 data set file.

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.

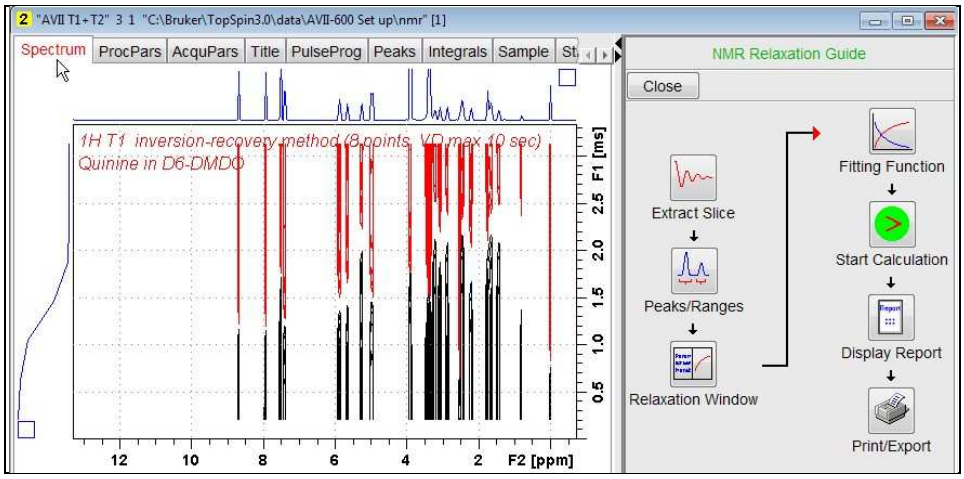
14) Provided phase constants have been correctly set up the transformed data set plot should resemble that shown below. Black = a positively phased signal, red = a negatively phased signal.



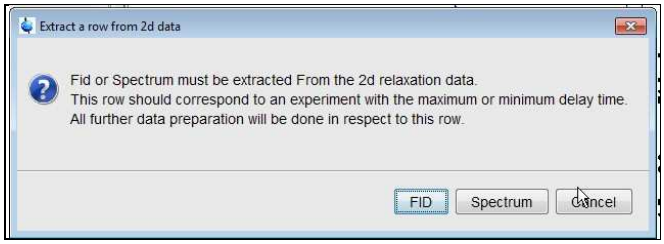
3.0 T_1 Data Set Processing

15) 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.

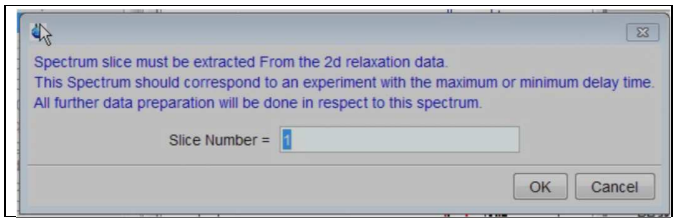
This will open up the screen display shown below.



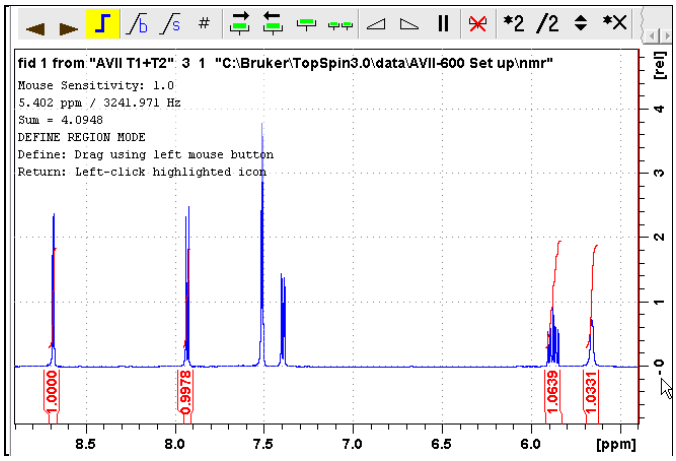
16) Click the **Extract Slice** button and then the click the **Spectrum** button in the panel that appears.



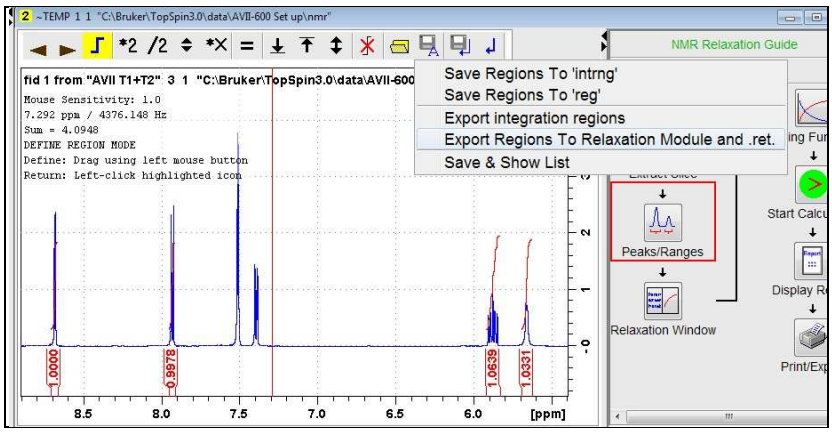
17) Enter **1** in the **Slice Number** cell and click the **OK** button. This will display the transformed spectrum ex the first (longest) **VDLIST** value = the one that was phased via the **rser 1** routine in steps 8 and 9.



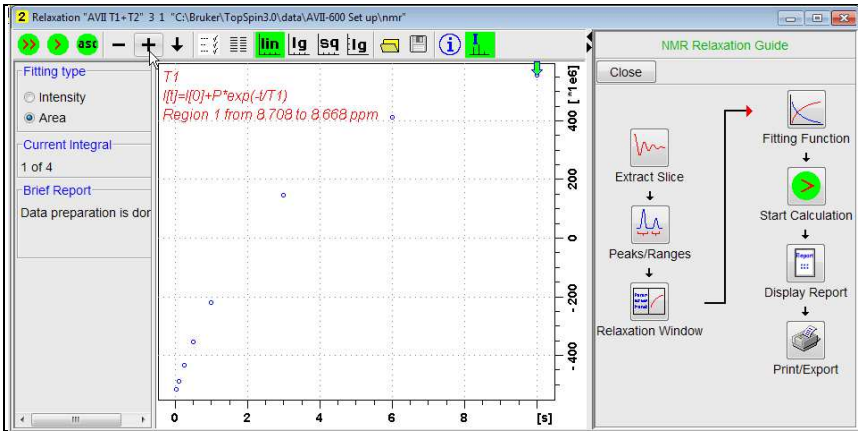
18) Expand the spectrum that appears using Topspin's "**E**" (Expand) menu button and integrate selected peaks in it as per standard ^{1}H spectrum processing. Integrals should start and terminate as close as possible to the edge of a peak.



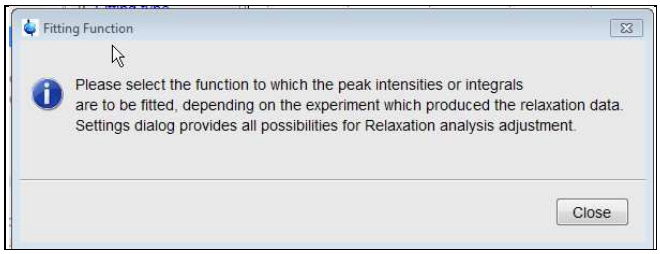
19) Click the "**Save Region As**" icon button (= 3rd from the right in TS3.2's the upper menu bar, or towards the left hand side of the menu bar in TS2.1) = *the one with the floppy disk icon + A below it*) and then its **Export Regions to Relaxation Module and .ret**. tab.



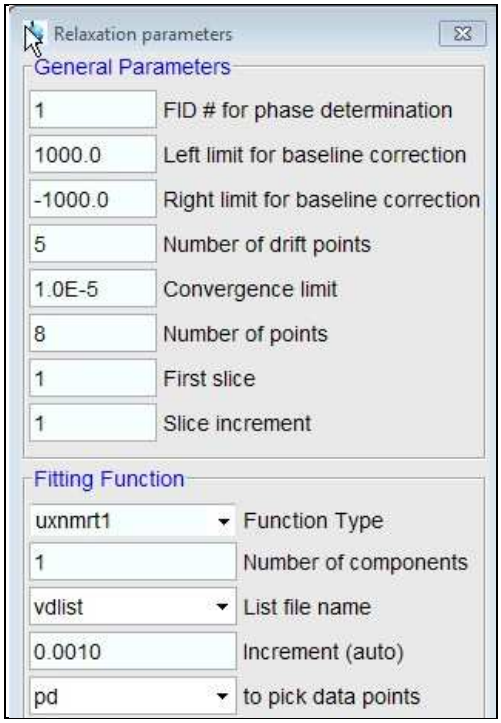
20) Click the **Relaxation Window** button, note any screen messages that may appear and close them. Select **Intensity** or **Area** in the plot window that appears. **Area** is often the better choice.

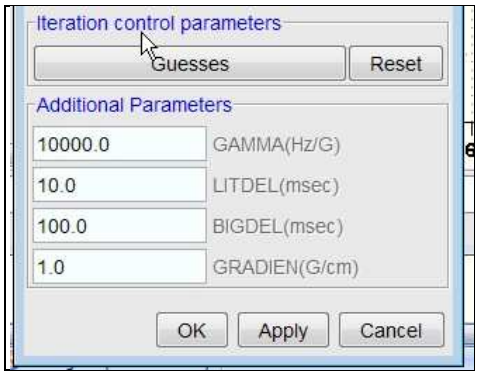


21) Click the **Fitting Function** and note the comments about **intensity** or **are**a options in the screen display that appears and close it.



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

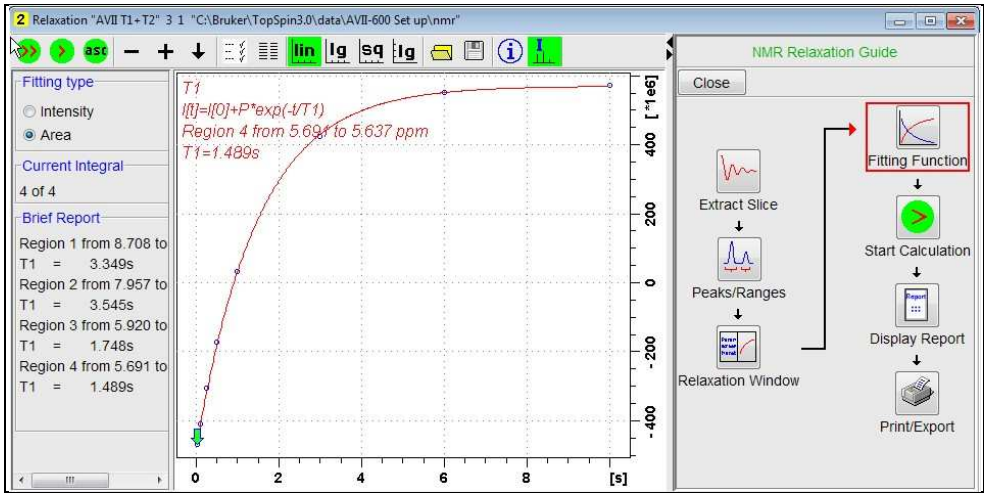




23) Check **Function Type = uxnmrt1** and **List file name = vdlist**.
Other cells/values can be left as they are (= default settings)

…. next page

24) 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.



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 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 :
 C:\Bruker\TopSpin3.0\data\AVII-600 Set up\nmr/AVII T1+T2/3/pdata/1
AREA fit :
 I[t]=I[0]+P*exp(-t/T1)

8 points for Integral 1,  Integral Region from 8.708 to 8.668 ppm
Results     Comp. 1

I[0]  =   1.093e+000
P     =   -2.024e+000
T1    =       3.349s
SD    =   1.021e-002

    tau    ppm     integral    intensity

   10.000s     8.678  5.5583e+008  6.5345e+007
    6.000s     8.678  4.1279e+008  4.7869e+007
    3.000s     8.678  1.4514e+008  1.4979e+007
    1.000s     8.678 -2.1925e+008 -3.0296e+007
  500.000m     8.678 -3.5543e+008 -4.7347e+007
  250.000m     8.678 -4.3437e+008 -5.7023e+007
  100.000m     8.678 -4.8692e+008 -6.3715e+007
   30.000m     8.678  -5.153e+008 -6.7561e+007
```