KJM 9250

ProtonT_1 Spectra on the AVIII800HD Spectrometer

Version 1.0

Topspin 3.5 Windows 7 AVIII HD 800

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1.0 Introduction

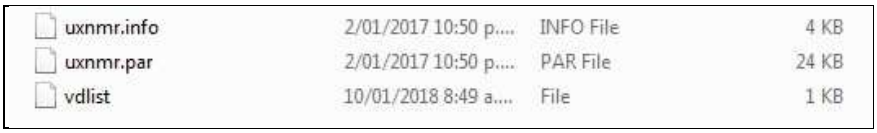
Three **aw coded protont1** parameter sets and linked **VDLIST files** have been set up on the **AVIIIHD-800** spectrometer running under **TopSpin3.5.**

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 *longest delay (10 sec) the first used VDLIST value and the shortest delay (0.03 sec) the last used value.*

The **awproton30t1** and **awproton60t1** parameter sets have **D1** and $\mathbf{VD_{max}}$ = 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.



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

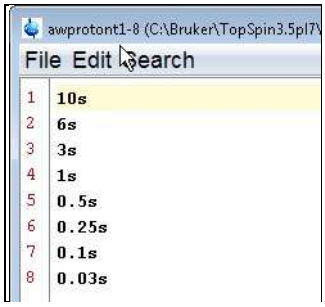
2.0 Experiment Set Up

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**

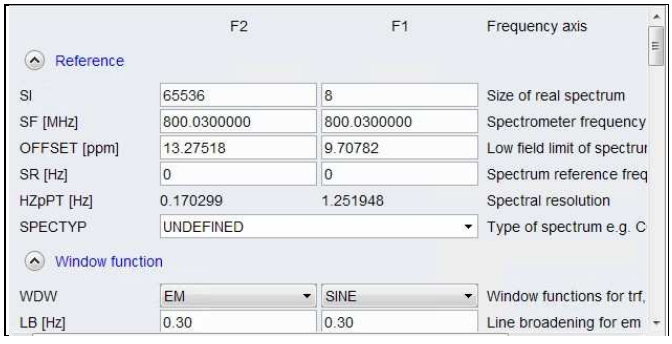
4) The **AWPROTONT1-8 VDLIST** file should have the entries shown below. Values are in seconds. Do not alter the values in this file. An alternatively named **VDLIST** file should be created if different **VD** and **D1** values are required for a particular compound.



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!).*

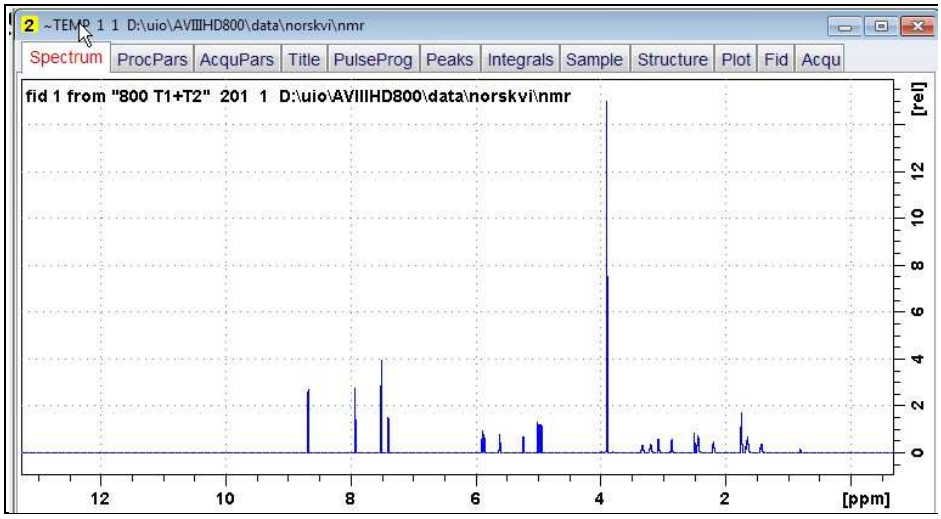
6) Type **edp** (enter) and check that **SI(F2) = 32 or 64K**, **SI(F1) = 8, WDW(F2) = EM, LB (F2) = 0.3-0.5 Hz** or other value of your choice. *Data sets with **12** or **16** **VDLIST** points are processed with **SI(F1) = 16.***



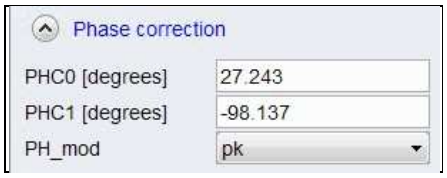
7) Start the acquisition using the **ZG** command.

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.

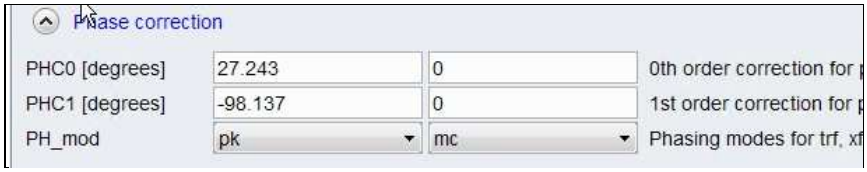


10) Type **edp** (enter) and note the phase constants for this spectrum.



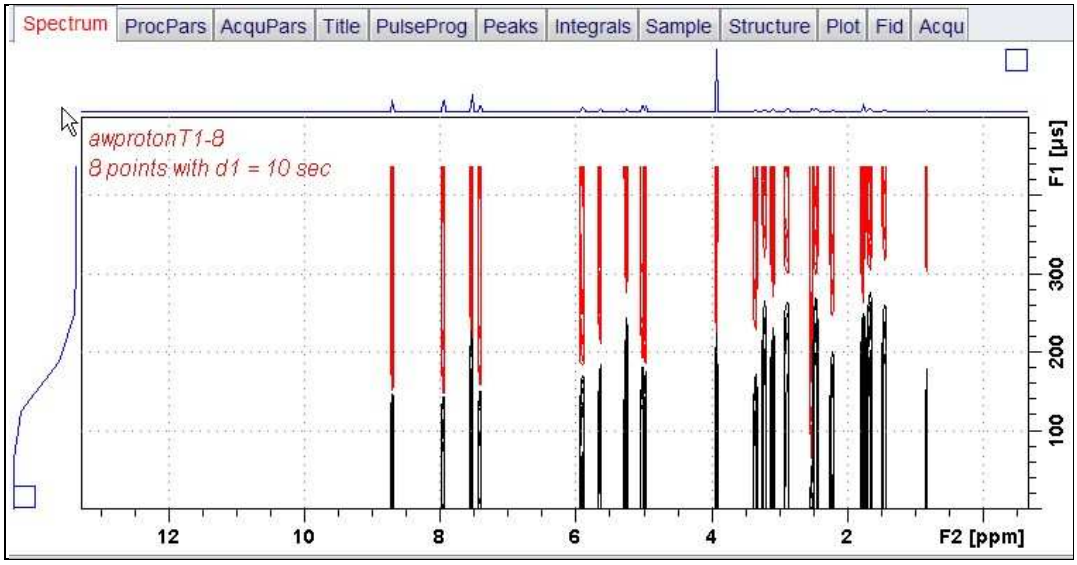
11) Close the **TEMP** window and reload the T_1 data set file.

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.



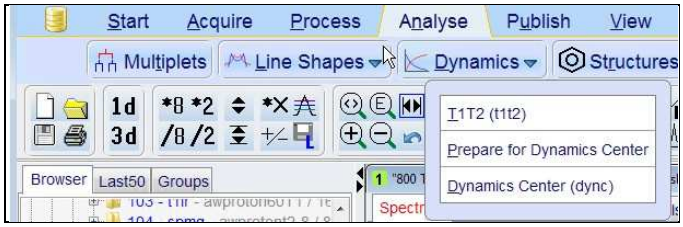
13) Type **xf2** (return) to transform the 2D data set followed by **abs2** (return) to baseline smooth it.

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.



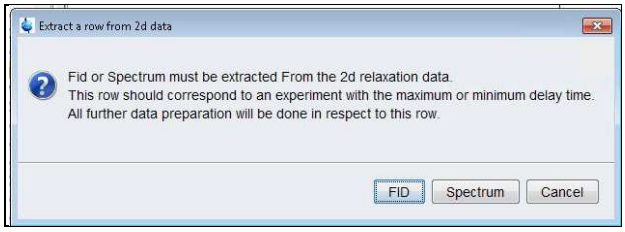
3.0 T_1 Data Set Processing

15) Click Topspin's **Analyse** menu tab then its **Dynamics s**ub-menu tab and click its **T1T2** tab.

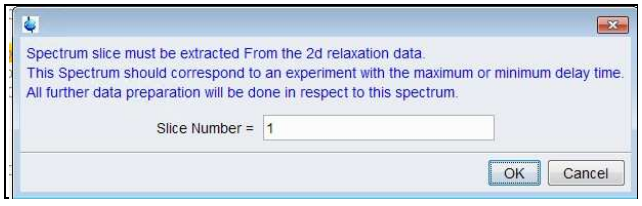


16) This will open the T1/T2 processing menu bar shown below.

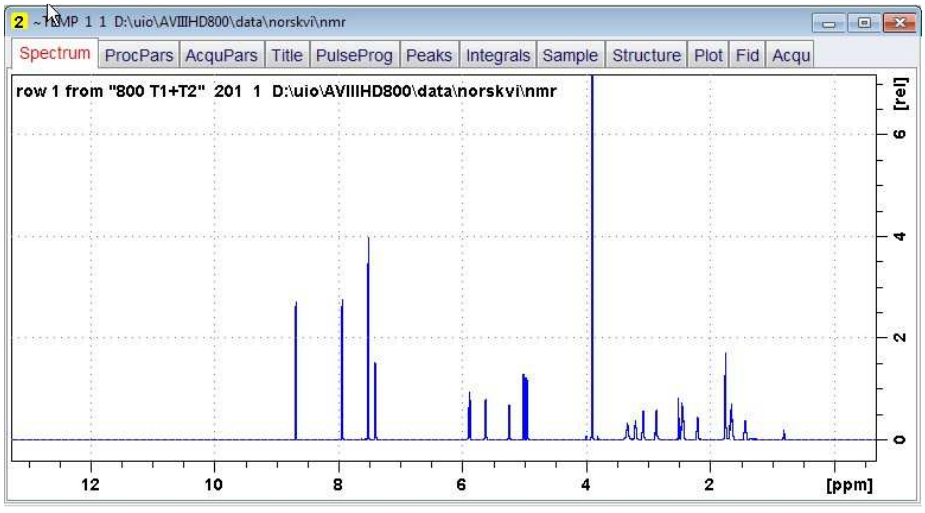
Click the **FID** tab and click the **Spectrum** button in the screen panel that opens.



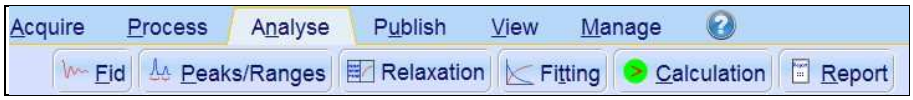
17) The screen panel shown below will appear**.**

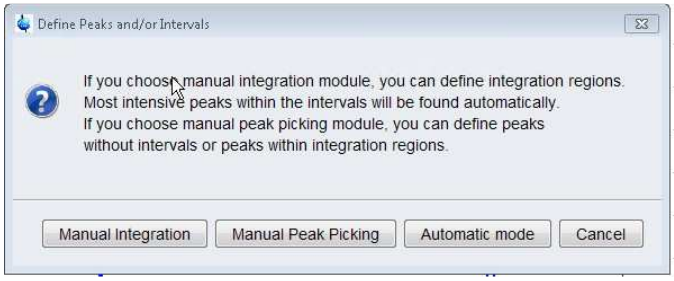


Enter **1** in the **Slice Number** cell and click the **OK** button. This will display the transformed spectrum ex the first (longest) **VDLIST** value = the one that was phased via the **rser 1** routine in steps 8 and 9.

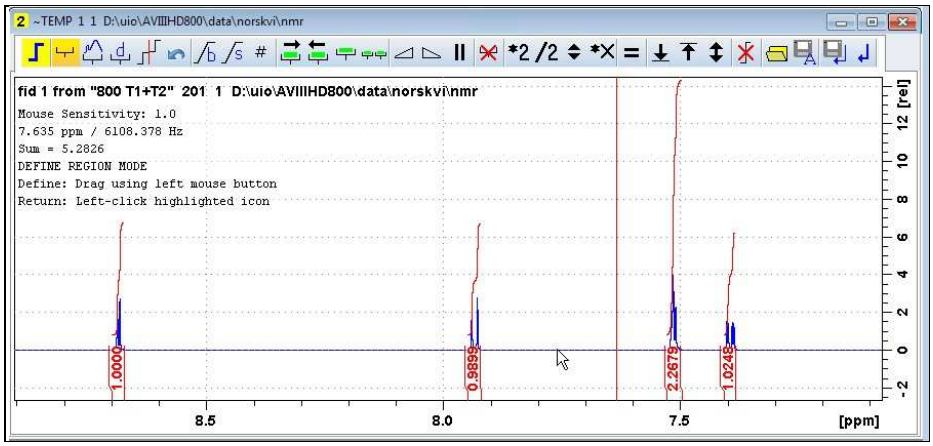


18) Expand the spectrum that appears in the usual way and click the **Peaks/Ranges** menu bar tab.





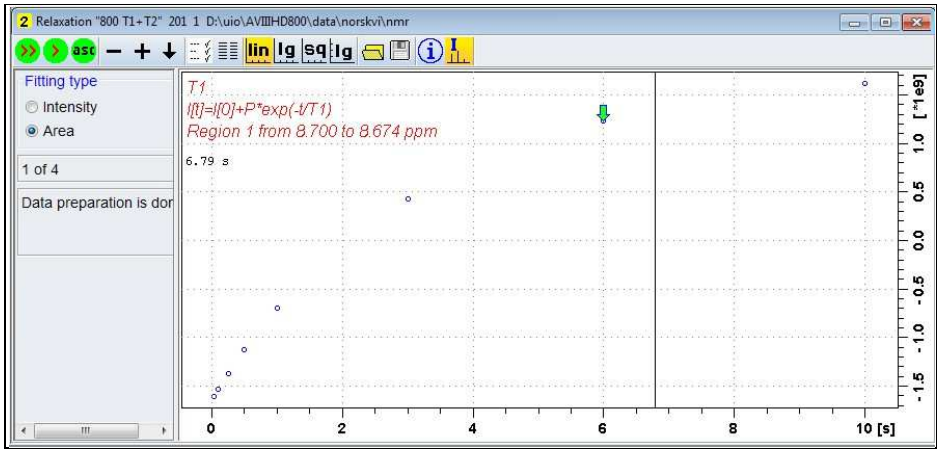
Select the **Manual integration** tab, close any screen messages that appear, and integrate selected peaks in the usual way.



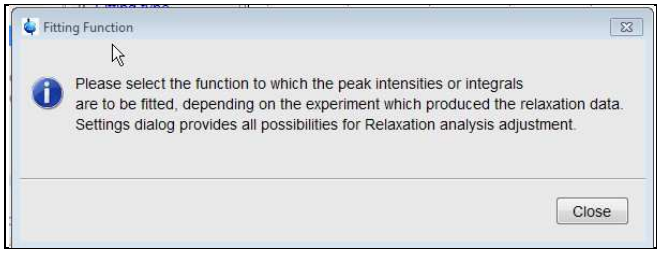
19) Click the "**Save Region As**" icon menu bar button (= *the floppy disk icon with A below it*) and then its **Export Regions to Relaxation Module and .ret**. tab.

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).

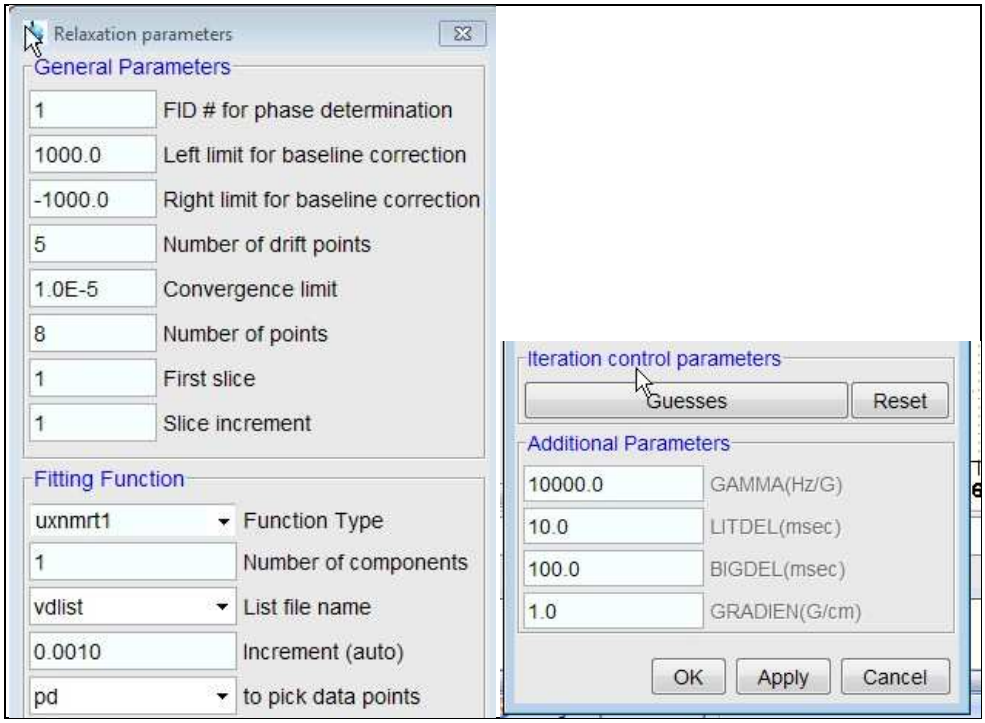
(plot window: → next page)



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

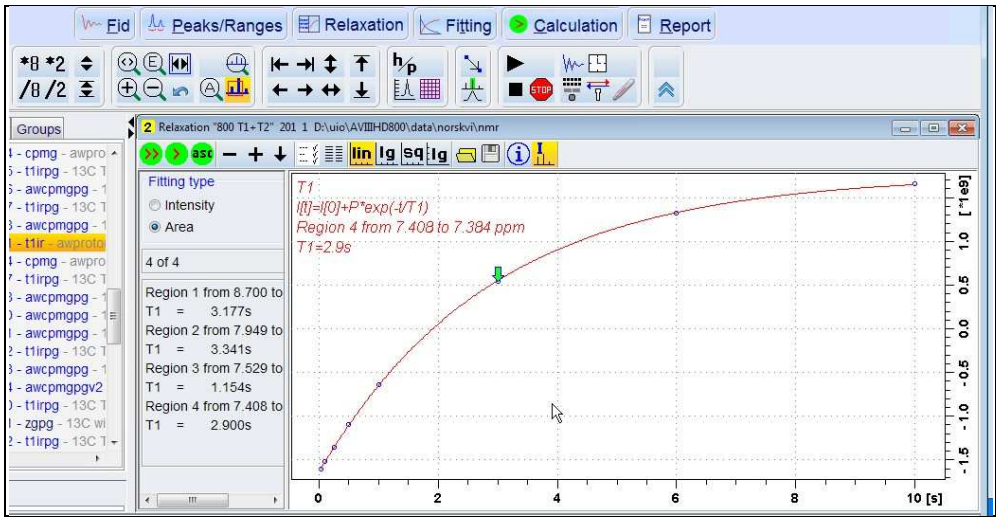


22) When the **Fitting** function message screen is closed a panel with **Relaxation parameters** (split into two screen captures below) will appear.



23) Check 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.



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 **Report** tab in the .menu bar can be used to view fitting report. A sample report for one signal is shown below.

Fitting report

File Edit Search

```
dataset :
 D:\uio\AVIIIHD800\data\norskvi\nmr/800 T1+T2/201/pdata/1
AREA fit :
 I[t]=I[0]+P*exp(-t/T1)

8 points for Integral 4,  Integral Region from 7.410 to 7.378 ppm
Results     Comp. 1

I[0]  =   1.062e+000
P     =   -2.043e+000
T1    =       2.897s
SD    =   3.402e-003

    tau    ppm     integral    intensity

   10.000s     7.401  1.7097e+009     5.2e+007
    6.000s     7.401  1.3686e+009  4.1392e+007
    3.000s     7.401  5.6961e+008  1.6667e+007
    1.000s     7.401 -6.5005e+008 -2.0877e+007
  500.000m     7.401 -1.1172e+009 -3.5464e+007
  250.000m     7.401 -1.3877e+009 -4.3569e+007
  100.000m     7.401 -1.5619e+009 -4.9122e+007
   30.000m     7.401  -1.649e+009 -5.1739e+007
```