KJM 9250

ProtonT_1 Spectra on the AVIII800HD Spectrometer

Version 5.0

Topspin 3.5 Windows 7 AVIII HD 800

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ProtonT$_1$ Spectra 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 _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 **VD$_{max}$** = 30 or 60 sec respectively and linked **VDLIST** files with **12** or **16** points respectively.

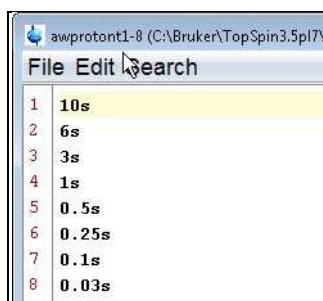**D1** and the longest (first) **VDLIST** value should be 3-5 times the longest T$_1$ in the sample compound.

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.


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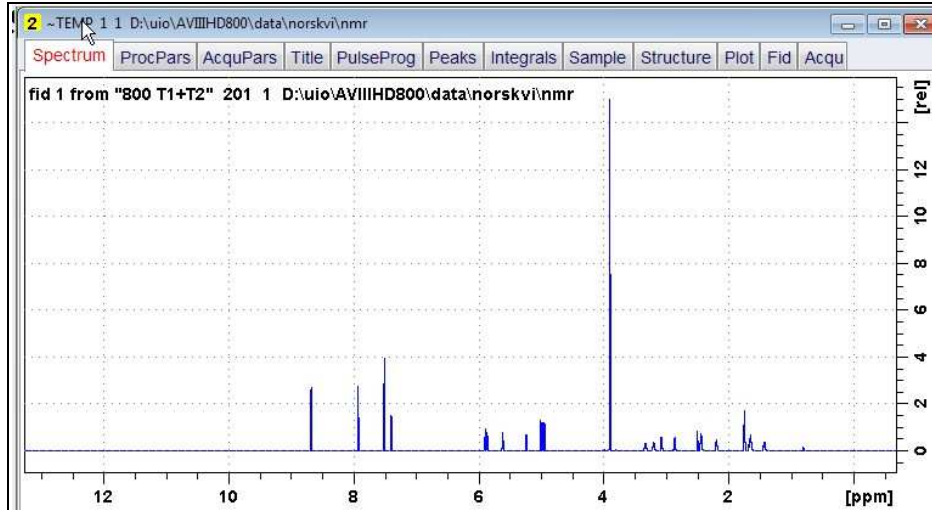
**10)** Type **edp** (enter) and note the phase constants for this spectrum.

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.
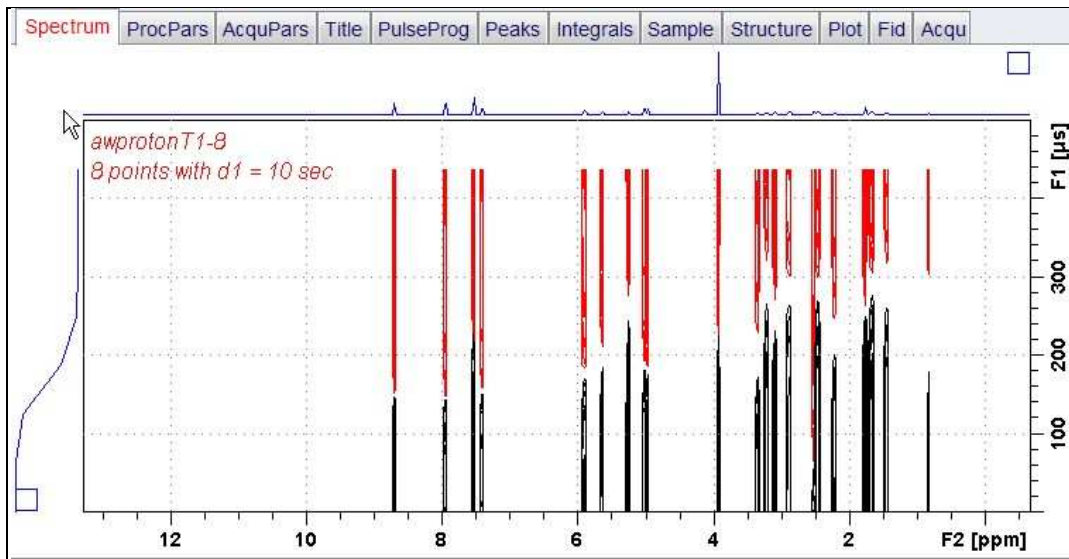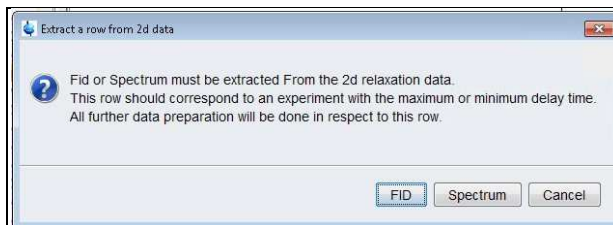
3.0   T₁ Data Set Processing

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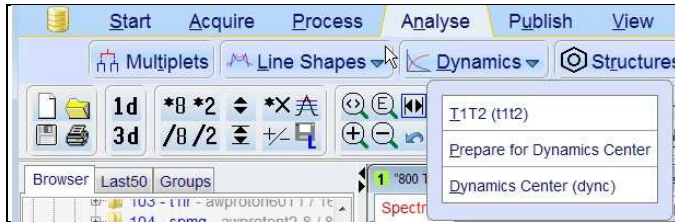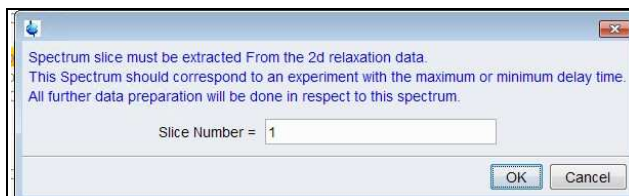

**16**) This will open the T1/T2 processing menu bar shown below.

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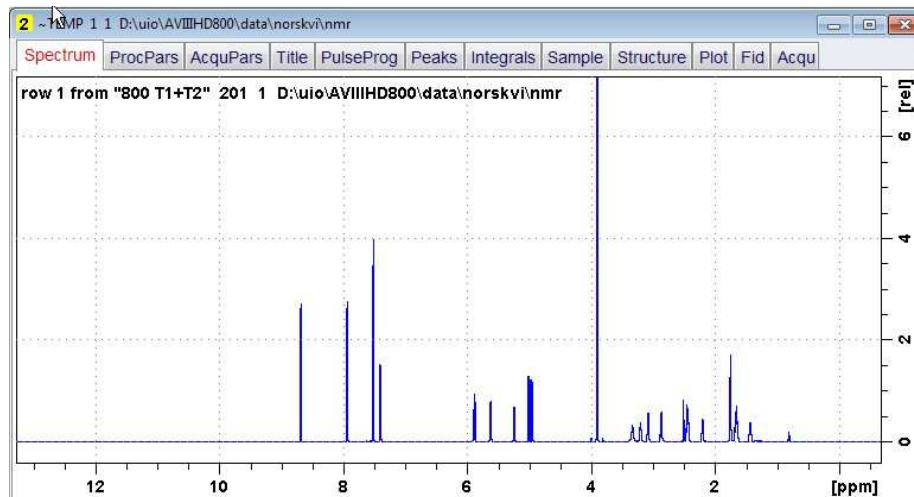
18) Expand the spectrum that appears in the usual way and click the **Peaks/Ranges** menu bar tab.
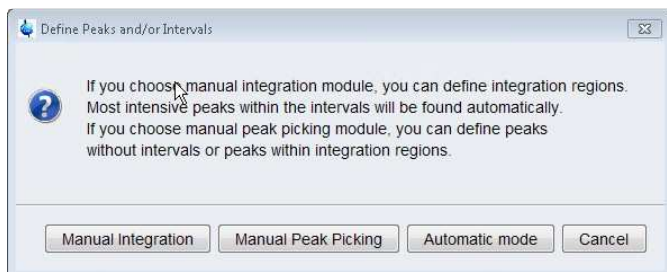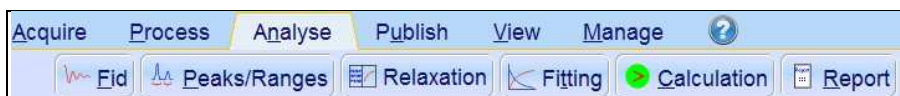

Select the **Manual integration** tab, close any screen messages that appear, and integrate selected peaks in the usual way.
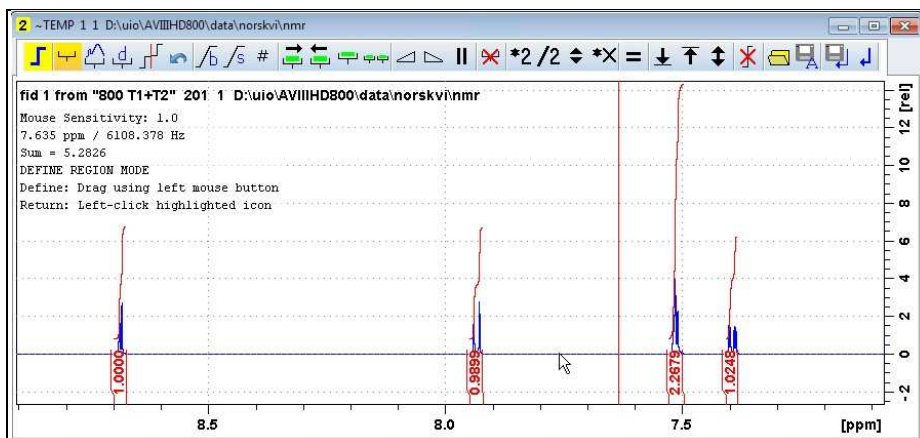
19) Click the "**Save Region As**" icon menu bar button (= *the floppy disk icon with A below it*) and then its **Export Regions to Relaxation Module and .ret**. tab.
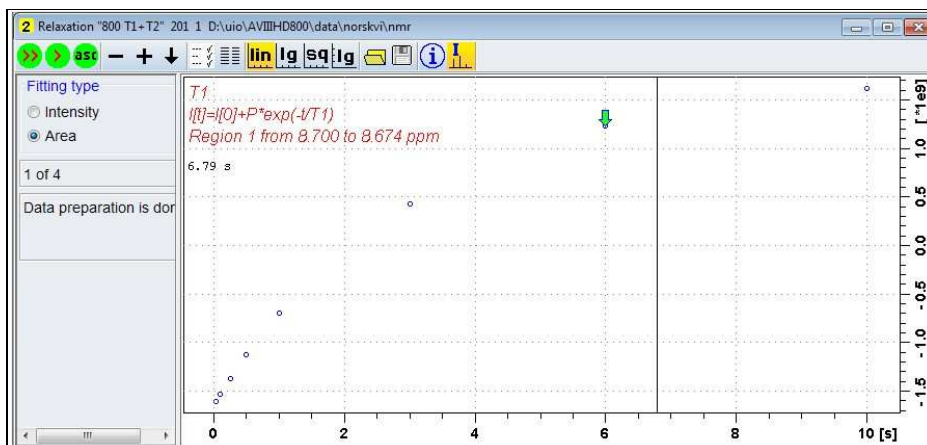

20) Click the **Relaxation** menu tab button, note any screen messages that may appear and close them. Select **Intensity** or **Area** in the plot window that appears. **Area** is invariably the better choice (next page).
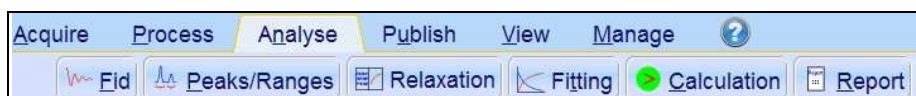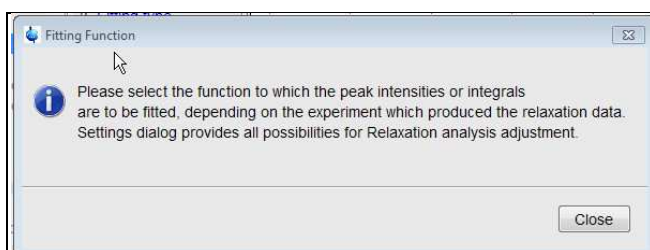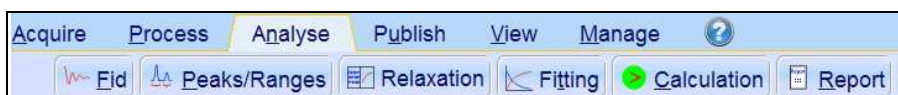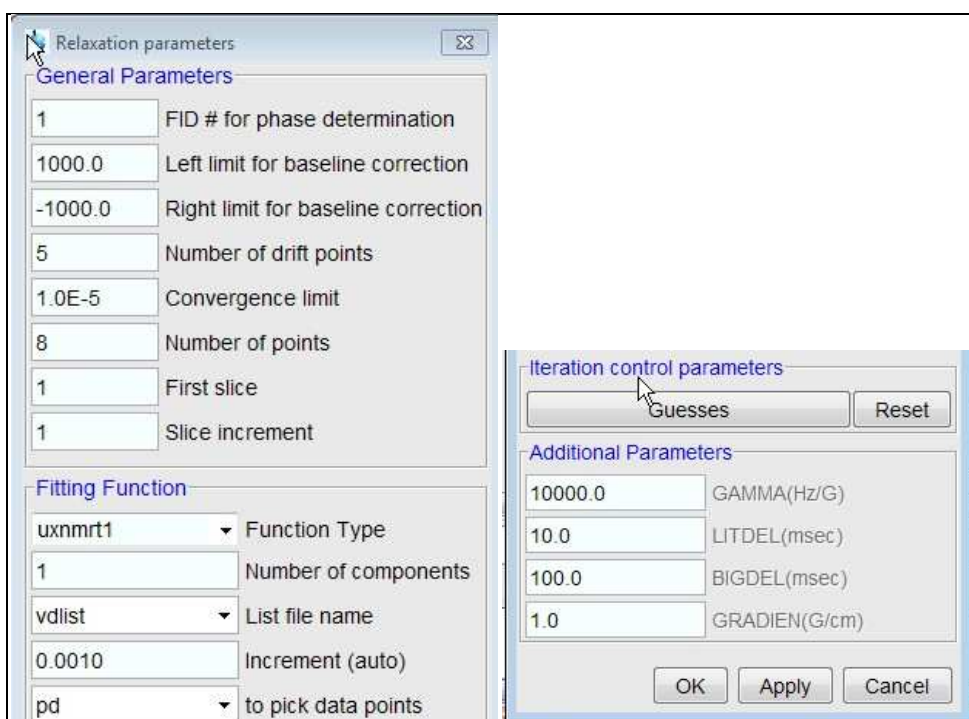

(plot window: → *next page)*

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
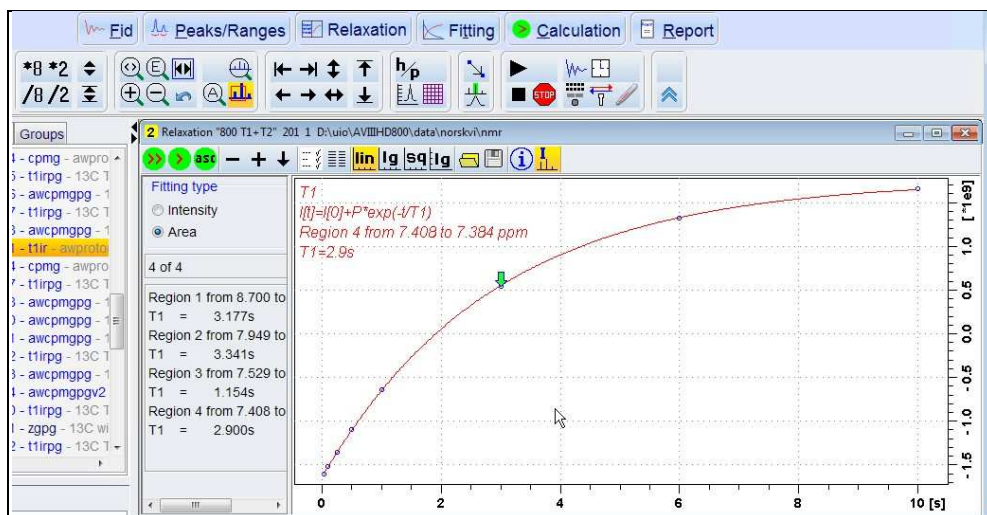

22) When the **Fitting** function message screen is closed a panel with **Relaxation parameters** (split into two screen captures below) will appear.
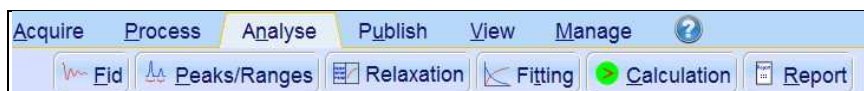
23) Check that **Function Type** = **uxnmrt1** and **List file name** = **vdlist**.

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.