KJM 9250

CarbonT_1 Spectra on the AVIII800HD Spectrometer

Version 1.0

Topspin 3.5 Windows 7 AVIII HD 800

April 2018

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1.0 Introduction

An aw coded **carbont1** parameter set with a linked **VDLIST file** has been set up on the **AVIIIHD-800** spectrometer running under **TopSpin3.5.**

The **awcarbont1** 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.

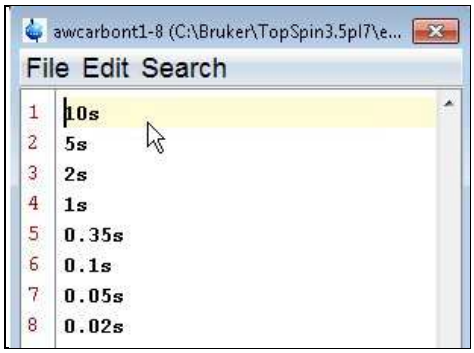
2.0 Experiment Set Up

1) Create an experiment and use the **rpar** command to read in the following parameter set: **awcarbont1** (d1 = 10 sec) + **getprosol**,
pulse programme = **t1irpg**.

2) Review default settings. These settings can be adjusted if required.
TD(F2) = 64K or 32K,**TD(F1) = 8**
SW = 240 ppm, O1P = 110 ppm.
D1 = 10 sec
NS = a **multiple of 2, 4 or 8, DS = 0, 2 or 4.**

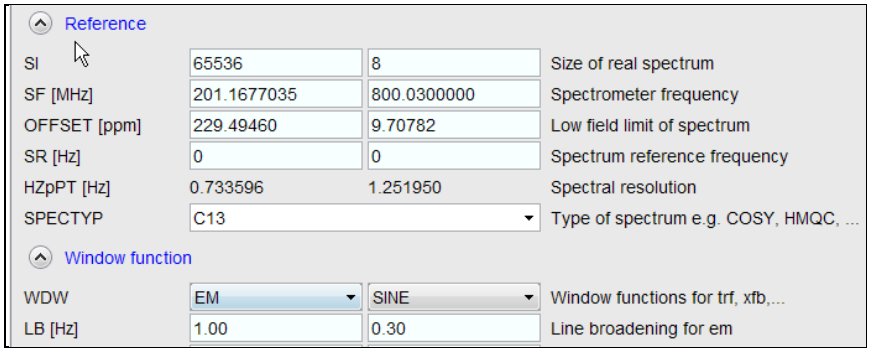
3) Type **ased** (enter) and review other parameters used in the job including the linked **VDLIST** file = **AWCARBONT1-8**

4) The **AWCARBONT1-8 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.



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

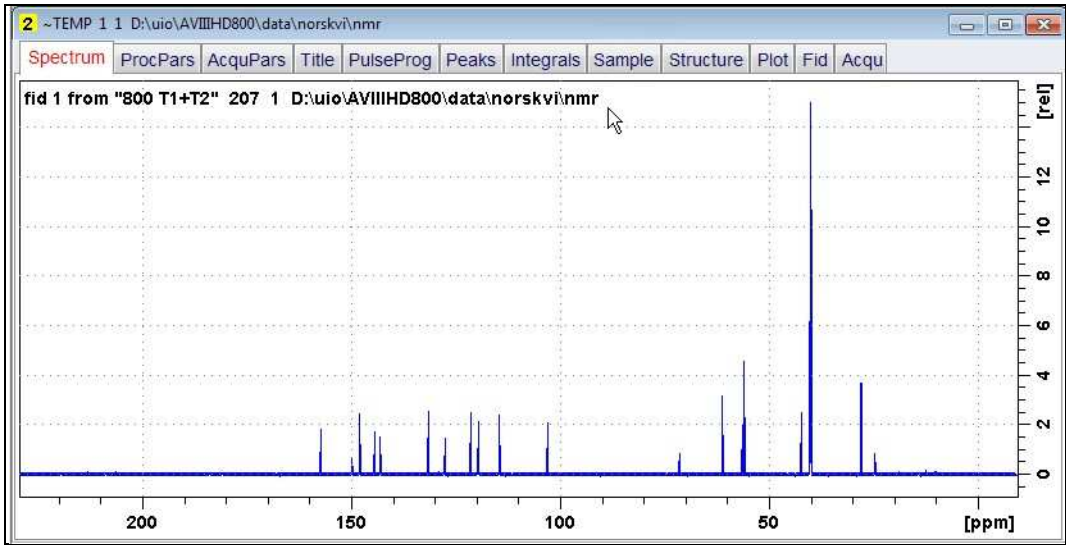
6) Type **edp** (enter) and check that **SI(F2) = 32 or 64K**, **SI(F1) = 8, WDW(F2) = EM, LB (F2) = 1.0-2.0 Hz** or other value of your choice.



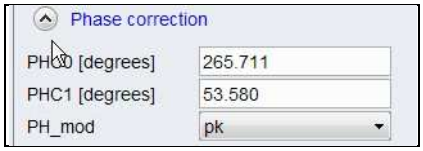
7) Start the acquisition using the **ZG** command.

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 carbon spectrum.

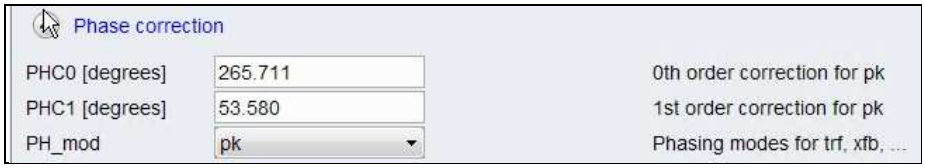


10) Type **edp** (enter) and note the phase constants for this spectrum.



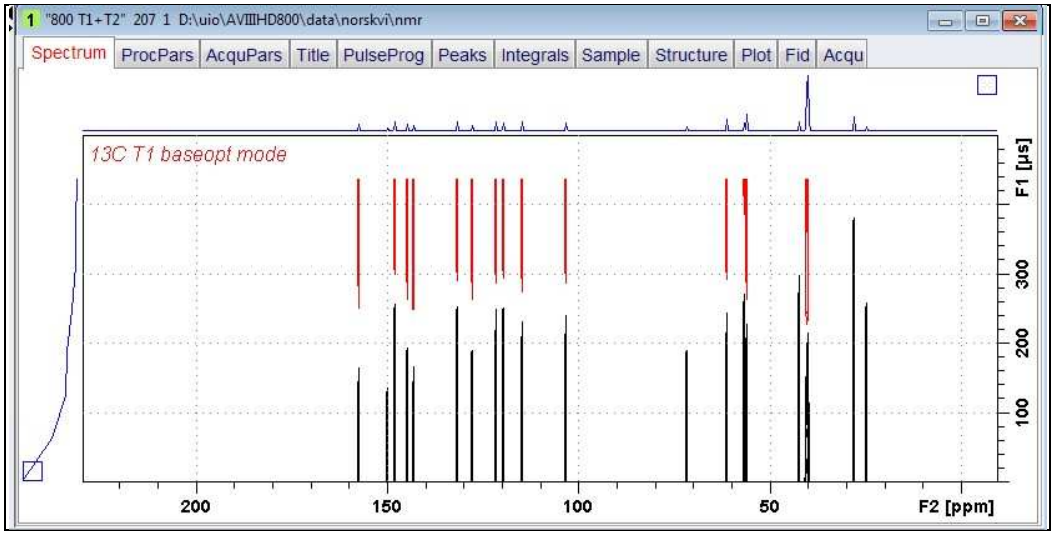
11) Close the **TEMP** window and reload the T_1 data set file.

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.



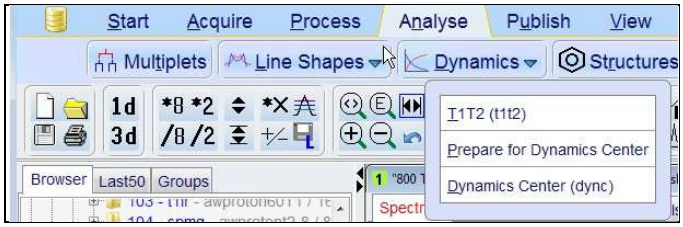
13) Type **xf2** (return) to transform the 2D data set followed by **abs2** (return) to baseline smooth it.

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.



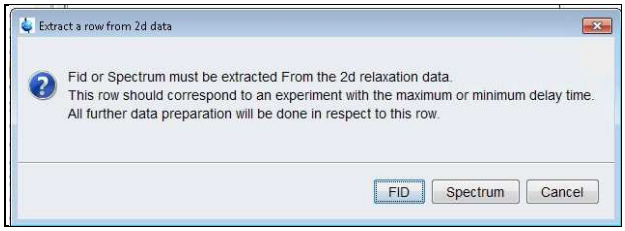
3.0 T_1 Data Set Processing

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

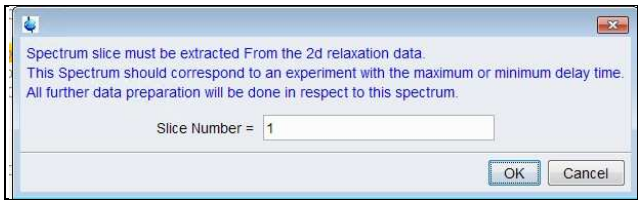


16) This will open the T1/T2 processing menu bar shown below.

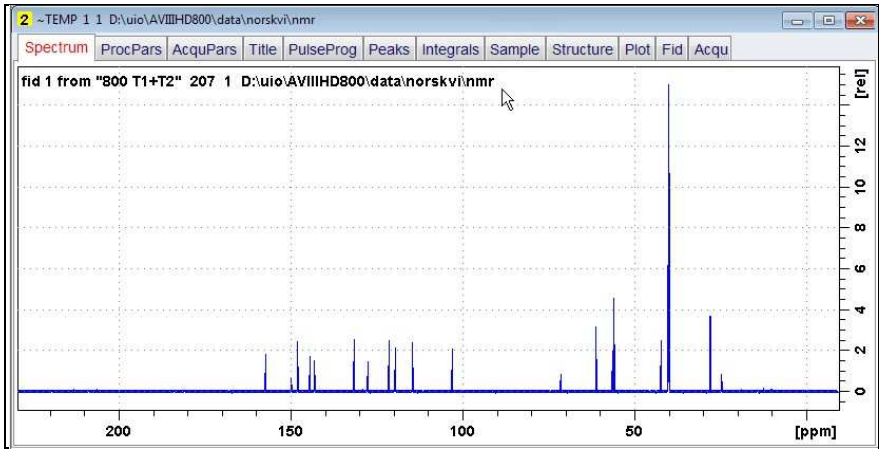
Click the **FID** tab and click the **Spectrum** button in the screen panel that opens.



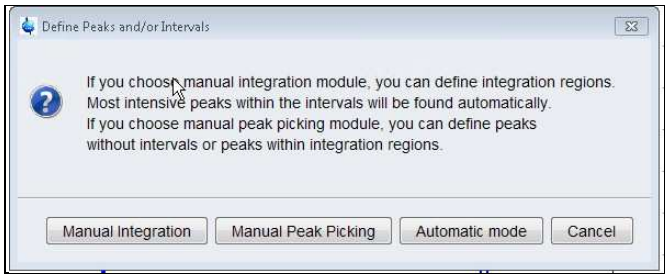
17) The screen panel shown below will appear**.**



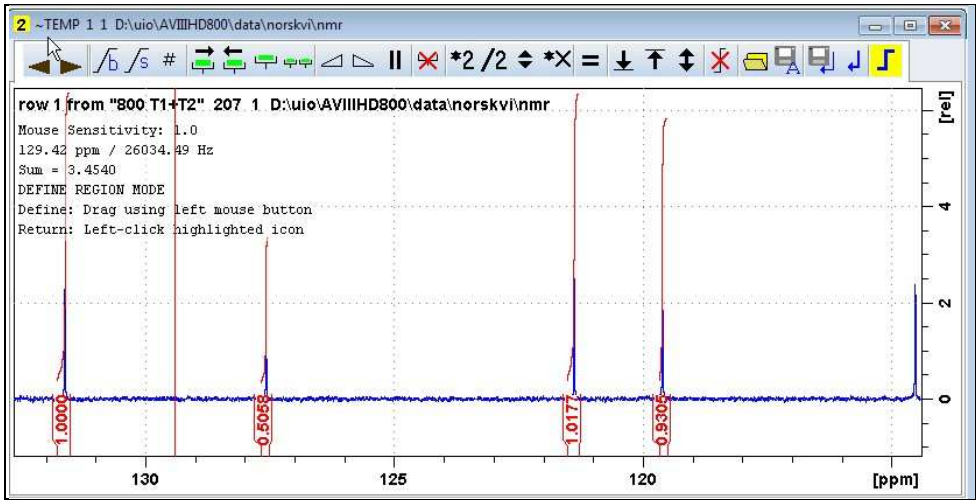
Enter **1** in the **Slice Number** cell and click the **OK** button. This will display the transformed spectrum ex the first (longest) **VDLIST** value = the one that was phased via the **rser 1** routine in steps 8 and 9.



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

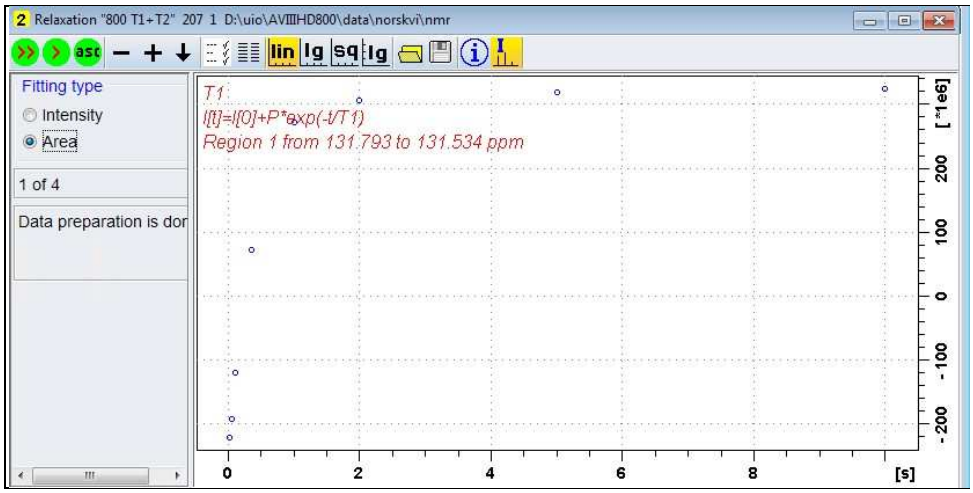


Select the **Manual integration** tab, close any screen messages that appear, and integrate selected peaks in the usual way .

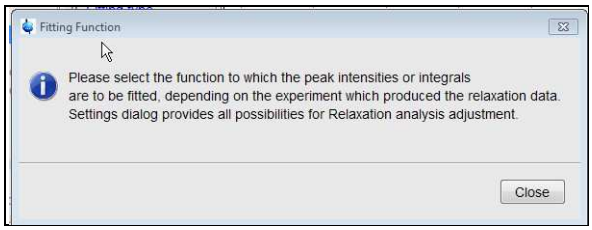


19) Click the "**Save Region As**" icon men bar button *(= the floppy disk icon with A below it*) and then its **Export Regions to Relaxation Module and .ret**. tab.

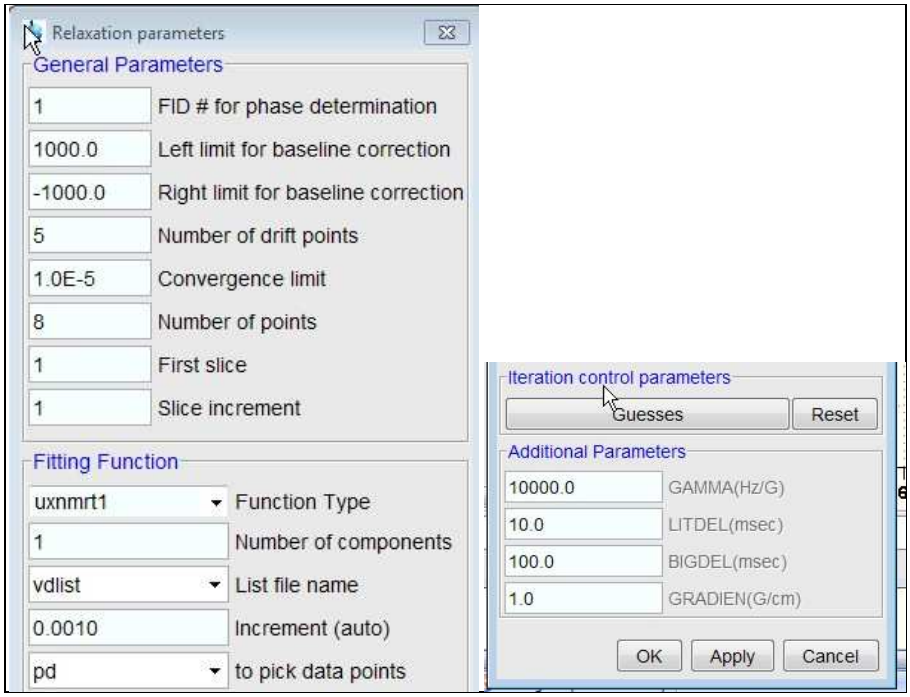
20) Click the **Relaxation** menu tab button, note any screen messages that may appear and close them. Select **Intensity** or **Area** in the plot window that appears. **Area** is invariably the better choice



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 - on the next page) 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 **NOT** 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.