KJM 9250

CarbonT$_2$ Spectra on the AVI600 and AVII Spectrometers

Version 5.0

Topspin 1.3 Windows XP AVI600
Topspin 2.1 Windows 7 AVII600

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Carbon T_2 Spectra on the AVI-600 and AVII-600

1.0 Introduction

An **awcarbont2** parameter set and linked **VCLIST** file from which a **VDLIST** file *must* be calculated prior to processing a T_2 data set have been set up on the AVI-600 and AVII-600 spectrometers running under TS2.1 and TS3.2 respectively.

The parameter sets have **D1 = 10 sec** and 8 x **VCLIST** values. The T_2 data set is acquired using the *smallest vclist value first. Prior to processing T_2 data sets variable constant (VC) values must be converted to time domain variable delay (VD) values using the* ***vctovd*** *command (see instructions 15 and 16.)*

D1 and the longest **VD** time derived from the largest VC **value** should be 3-5 times the longest T_2 in the sample compound. T_2 is always shorter than T_1.

$$VD_{max} = [P2 + (2*D20)]* VC_{max}$$

T_2 data sets can be processed on the spectrometer terminal or off line using any version of Topspin provided the linked **VCLIST** file is either resident in the experiment's top level folder (= default setting: see below) or recreated or copied into the off line terminal's *C:\Bruker\TopspinX.X\exp\stan\nmr\lists\vc* folder.

If required the VCLIST file that is present in the experiment's top level folder can be opened with **WordPad** and the **VC** values in it can be viewed and used to recreate the original named **VCLIST** file from which the **VDLIST** file can be created using the **vctovd** command.

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

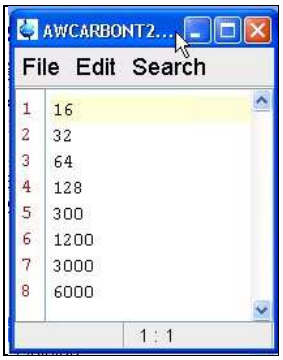
2.0 Experiment Set Up

1) Create an experiment and read in the **awcarbont2** parameter set **(+ getprosol).** Pulse programme = **awcpmgpg**

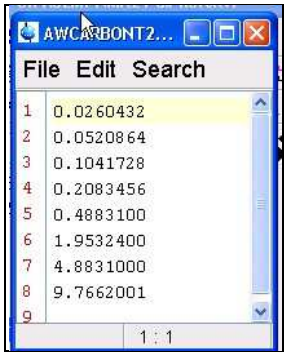
2) Review default settings. These settings can be adjusted if required.
TD(F2) = 32K, TD(F1) = 8
SW = 240 ppm, O1P = 110 ppm.
D1 = 10 sec.
D20 = 800 usec = 0.000800 sec
NS = multiple of 4 or 8, DS = 4 or 8.

3) Type **ased** (enter) and review other parameters used in the job including the linked **VCLIST** file **= AWCARBONT2-8.**

4) The **VCLIST** file should have the entries shown below. Do not alter the values in this file. An alternatively named **VCLIST** file should be created if different **VC** and **D1** values are required for a particular compound. The **VD** values calculated from the **AWCARBONT2-8** file's **VC** values using the **vctovd** command (see steps **15** and **16**) when **P2** = (180 degree pulse) = **27.7 usec** on AVI-600 and **D20** = **800 usec** = 0.0008 sec are shown below.



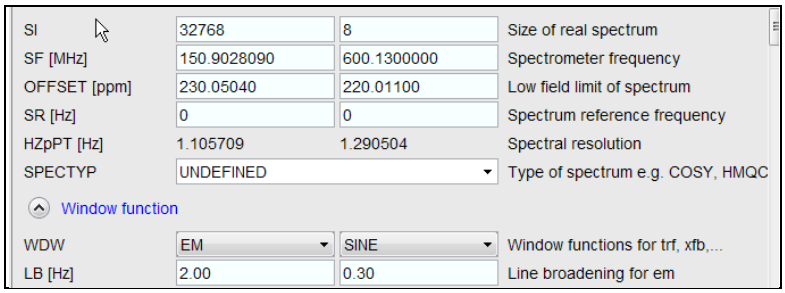
AWCARBONT2-8 VC file values



Calculated VD times (sec)

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

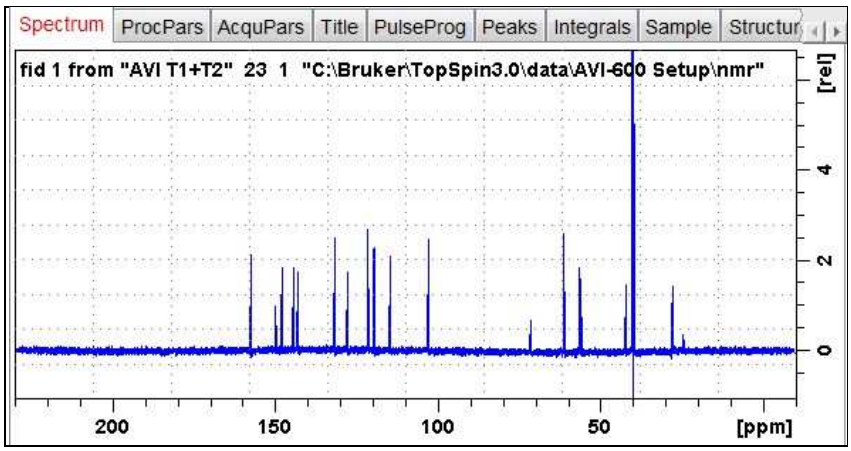
6) Type **edp** (enter) and check that **SI(F2) = 32K**, **SI(F1) = 8, WDW(F2) = EM, LB (F2) = 2 Hz** or other value of your choice.



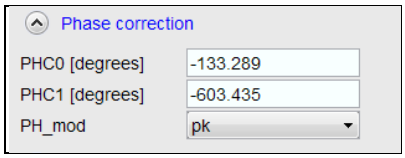
7) Start the acquisition using the **ZG** command

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.

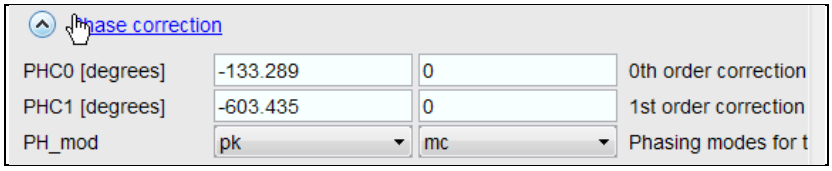


10) Type **edp** (enter) and note the phase constants for this spectrum. As per AVI and AVII ^{13}C spectra a large negative PHC1 value will be require



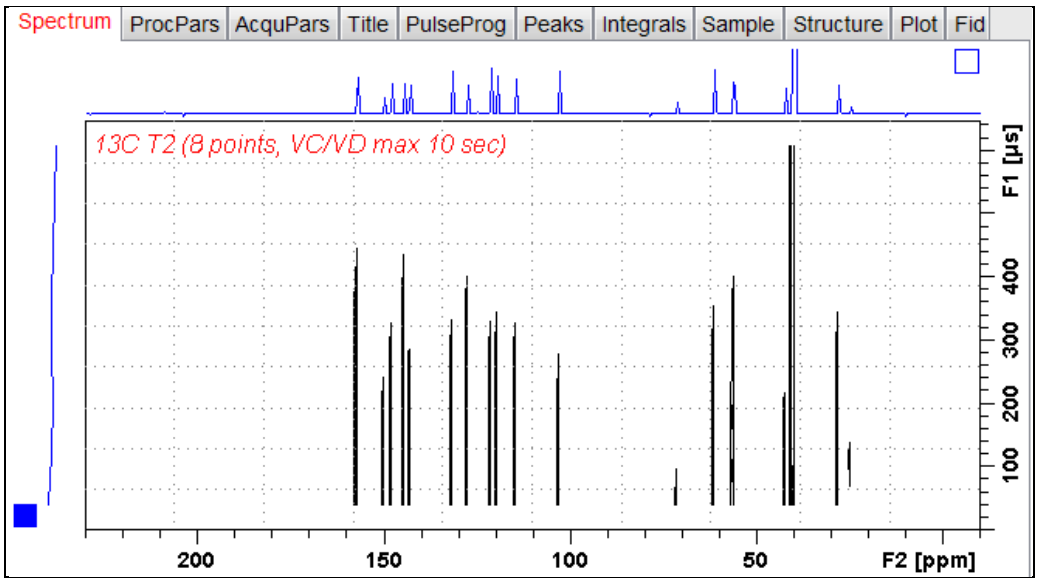
11) Close the **TEMP** window and reload the T_2 data set file.

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.

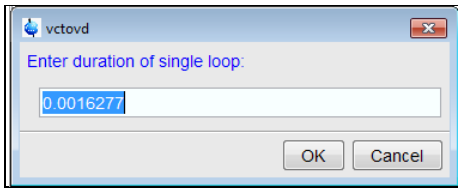
14) Provided phase constants have been correctly set up the transformed data set plot should resemble that shown below. Signals intensities go from their maximum positive value to zero intensity.



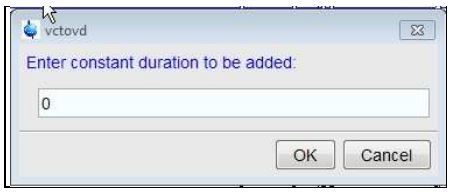
3.0 T_2 Data Set Processing

15) *Prior to processing a T_2 data set it is essential that variable constant (VC) values are converted to time domain variable delay (VD) values.*

16) Type **vctovd** (enter) and click OK on the display panels that appear.

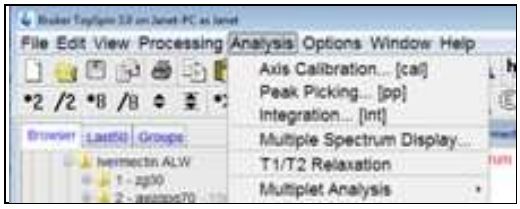


Single VC loop value = p2 + 2 x d20



Not used. .

17) 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.

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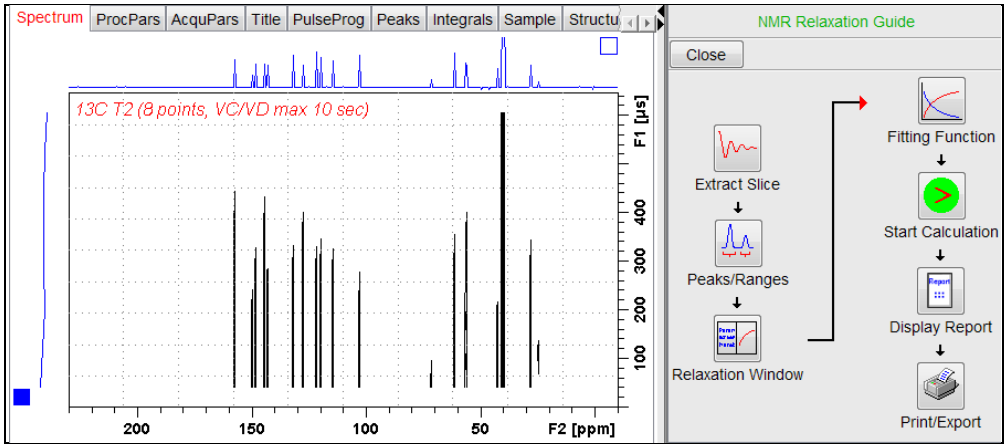
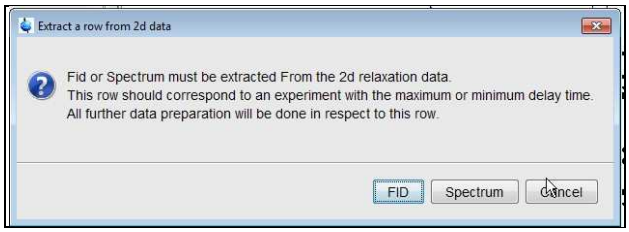
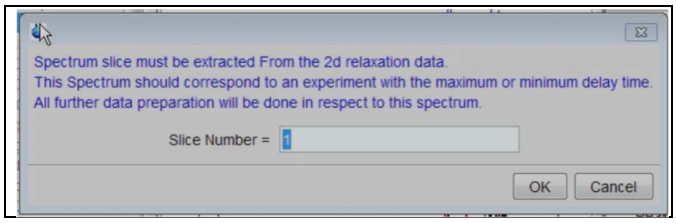


18) Click the **Extract Slice** button and then the click **Spectrum** button in the panel that appears.

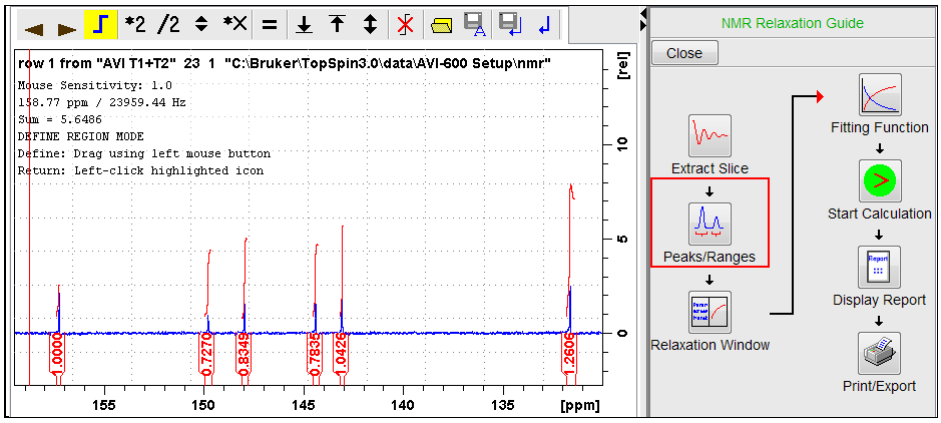


19) Enter **1** in the **Slice Number** cell and click the **OK** button. This will display the transformed spectrum ex the first (shortest) **VCLIST** → **VDLIST** value = the one that was phased via the **rser 1** routine in steps 8 and 9.

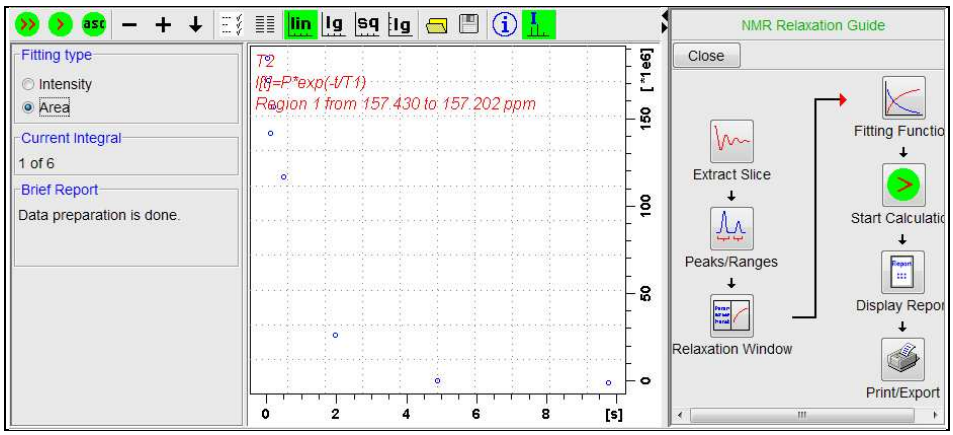


20) Expand the spectrum that appears in the usual way and integrate selected peaks in it. Integrals should start and terminate as close as possible to the edge of a peak.

21) 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..

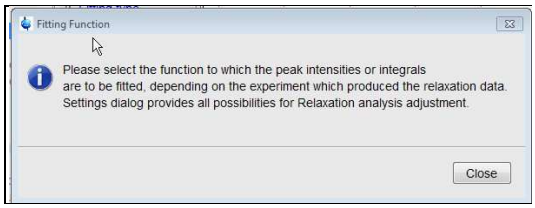


22) Click the **Relaxation Window** button and select **Intensity** or **Area** in the plot window that appears. **Area** is the best choice for carbon T_2 values.

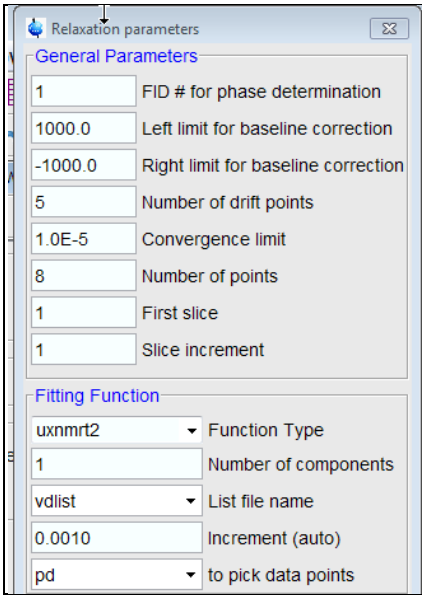


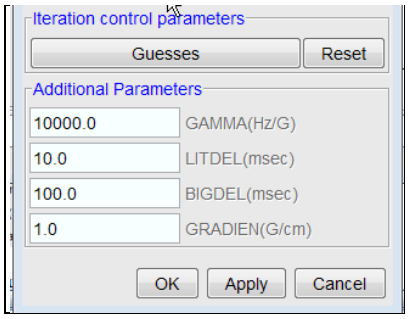
. *<u>The plot's upper title line correctly has T_2, but the T_1 equation incorrectly appears below it. This is a long standing Topspin bug.</u>*

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



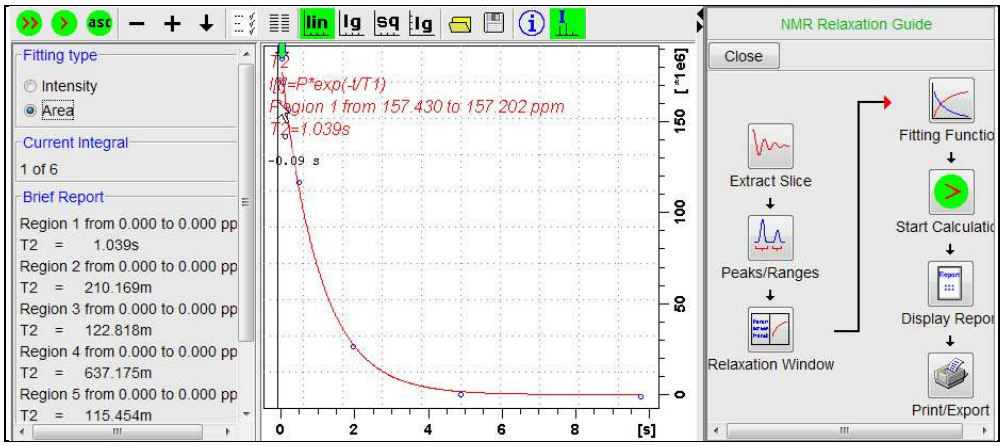
24) When the Fitting Function message screen is closed a panel with relaxation parameters (split into two screen captures below) will appear.





25) Check **Function Type = uxnmrt2** *(important!)* and **List file name = vdlist**.
Other cells/values can be left as they are (= default settings).
Click **Apply** and **OK** and close the window.

26) Click the **double red arrow** in the menu bar at the top left of the plot window and **NOT** the single red Start Calculation arrow button in the NMR Relaxation Guide menu below the Fitting Function button.



The appearance of the T_1 equation in this screen is a long standing Topspin error.

27) The + and - buttons in the upper menu bar can be used move through the series of T_2 plots.

28) 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_2 results and update their plots.

29) The NMR Relaxation Guide has buttons which can be used to display and/or print T_2 results. A sample report for one signal is shown below. Zero value points (= completely T_2 relaxed) or negative artifact peaks will be eliminated.

```
Dataset :
 C:\Bruker\TopSpin3.0\data\AVI-600 Setup\nmr/AVI T1+T2/23/pdata/1
AREA fit :
 I[t]= P*exp(-t/T2)

8 points for Integral 1,  Integral Region from 0.000 to 0.000 ppm
Results     Comp. 1

P     =   9.816e-001
T2    =       1.039s
SD    =   4.819e-002

    tau    ppm     integral    intensity

   26.043m   157.226  1.8464e+008  4.3523e+007
   52.086m   157.225  1.7182e+008  4.3054e+007
  104.173m   157.224  1.4193e+008  4.2111e+007
  208.346m   157.226  1.5655e+008  3.6103e+007
  488.310m   157.226  1.1662e+008  2.8708e+007
    1.953s   157.225  2.6262e+007  8.0794e+006
    4.883s   157.232 -2.8243e+005     eliminated
    9.766s   157.232 -1.0611e+006     eliminated
```