KJM 9250

CarbonT$_1$ Spectra on the DRX500 Spectrometer

Version 5.0

Topspin 1.3 Windows XP DRX 500

Carbon T_1 Spectra on the DRX-500 Spectromter

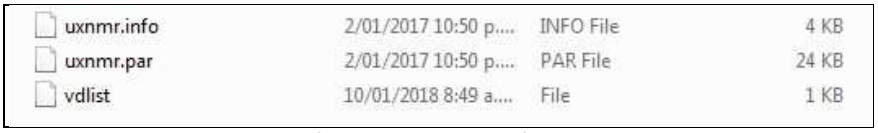
1.0 Introduction

An **awcarbont1** parameter set and linked **VDLIST** file have been set up on DRX-500 spectrometer running under **TS1.3.**

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.

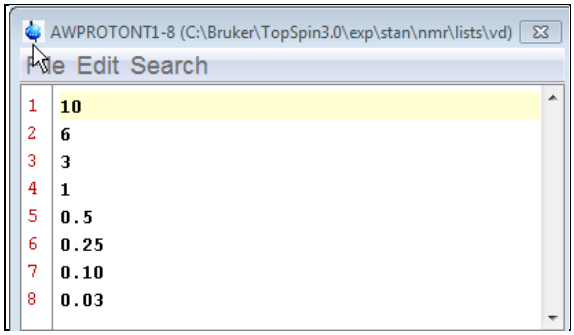
2.0 Experiment Set Up

1) Create an experiment and read in the **awcarbont1** parameter set **(+ getprosol).** Pulse programme = **t1irpg**

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

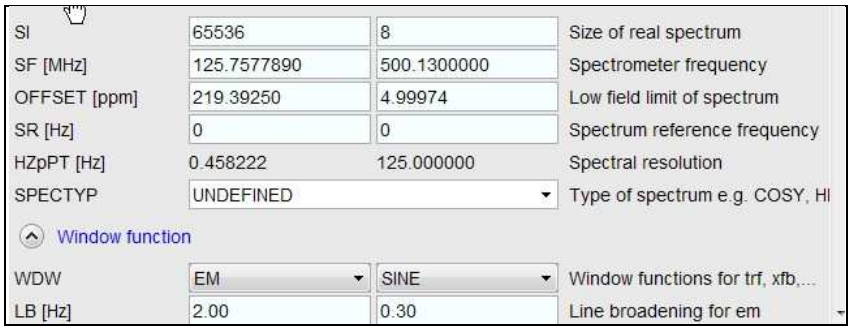
3) Type **ased** (enter) and review other parameters used in the job including the linked **VDLIST** file = **AWCARBONT1-8**

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!).*

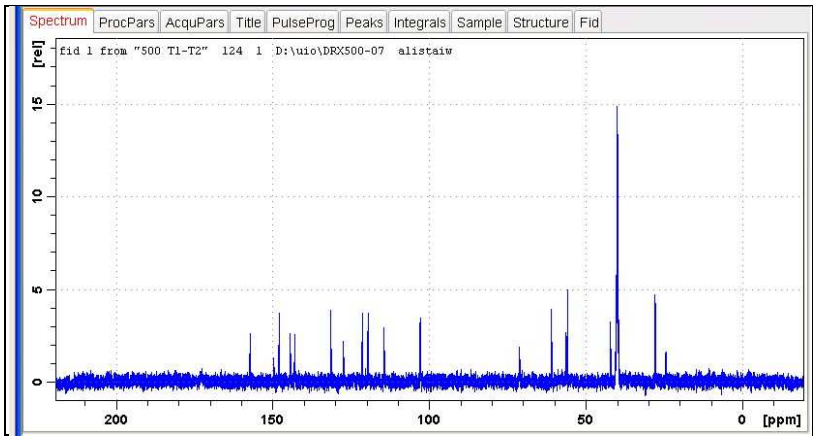
6) Type **edp** (enter) and check that **SI(F2) = 32K**, **SI(F1) = 8,**
WDW(F2) = EM, LB (F2) = 2 Hz or other value of your choice.



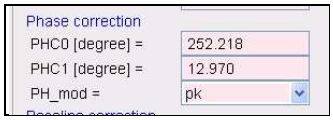
7) Start the acquisition using the **ZG** command.

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. The **multiabsn** command with **n = 30-40** can be used to straighten the baseline.

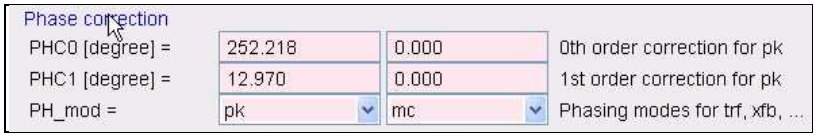


10) Type **edp** (enter) and note the phase constants for this spectrum.



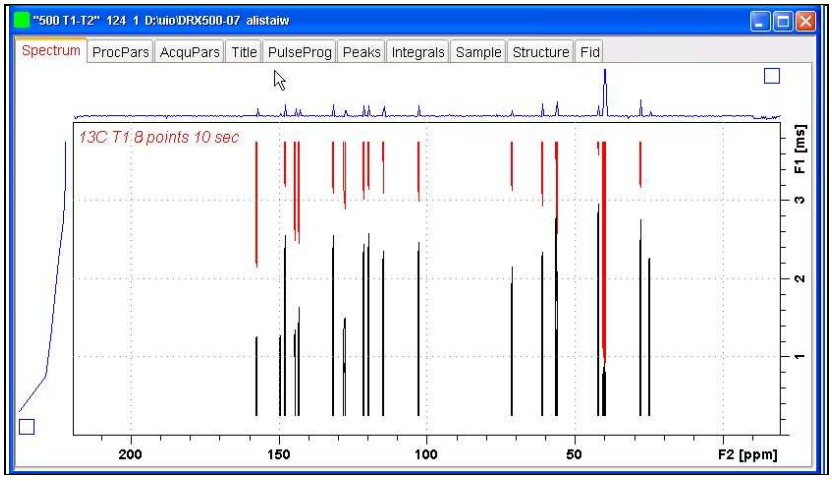
11) Close the **TEMP** window and reload the T_1 data set file.

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.



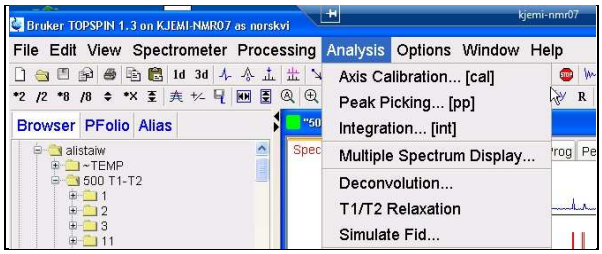
13) Type **xf2** (return) to transform the 2D data set followed by **abs2** (return) to baseline smooth it.

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.

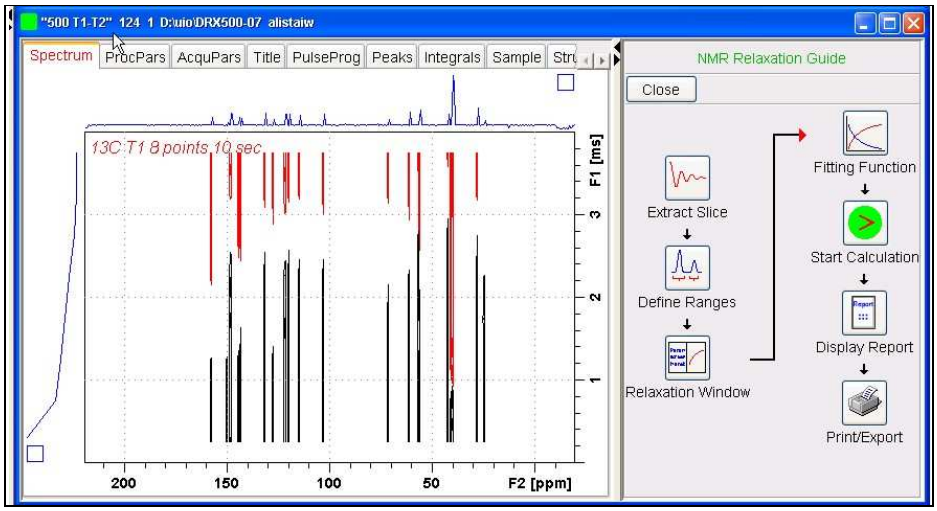


3.0 T_1 Data Set Processing

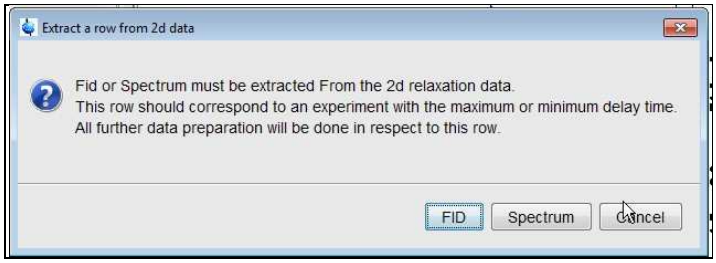
15) Open Topspin's **Analysis** menu and click its **T1/T2 Relaxation** tab.



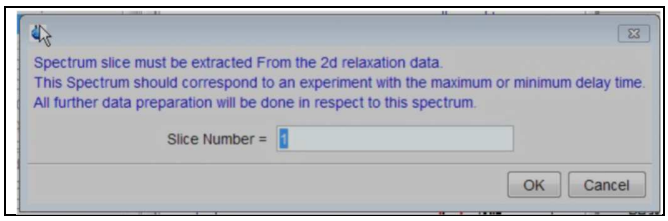
This will open up the screen display shown below.



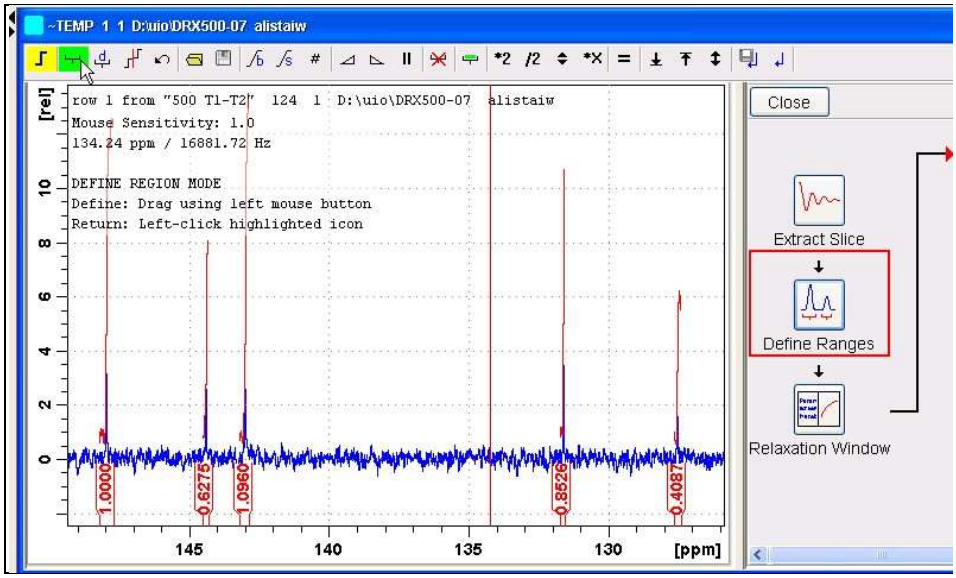
16) Click the **Extract Slice** button and then the click the **Spectrum** button in the panel that appears.



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 7 and 8.

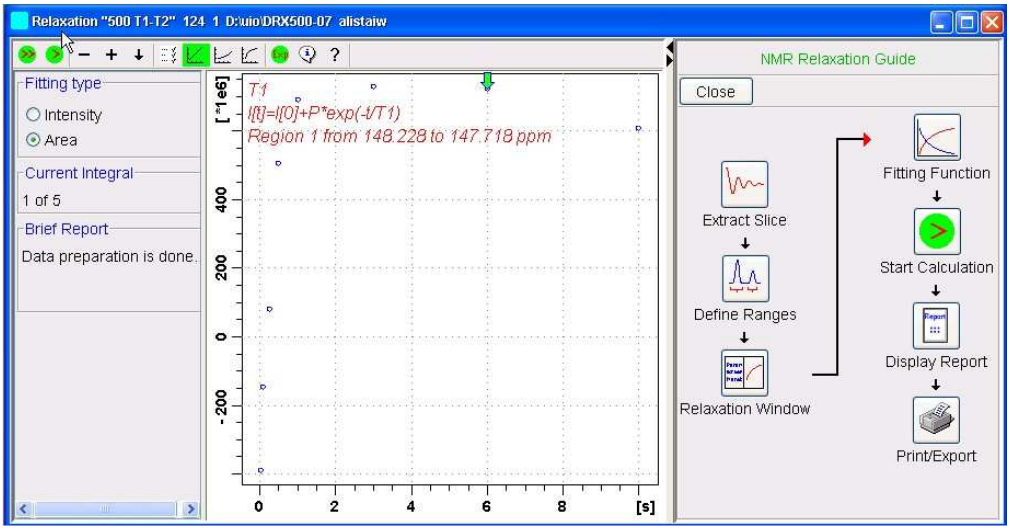


18) Expand the spectrum that appears in the usual way and integrate selected peaks in it. Integrals should start and terminate as close as possible to the edge of a peak.

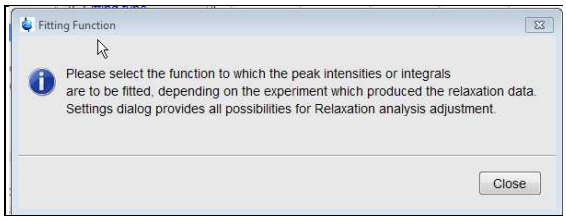


19) Click the "**Save Region As**" icon button (= 7th from the left in TS1.3's upper menu bar = *the one with the floppy disk icon*) and then its **Export Regions to Relaxation Module and .ret**. tab.

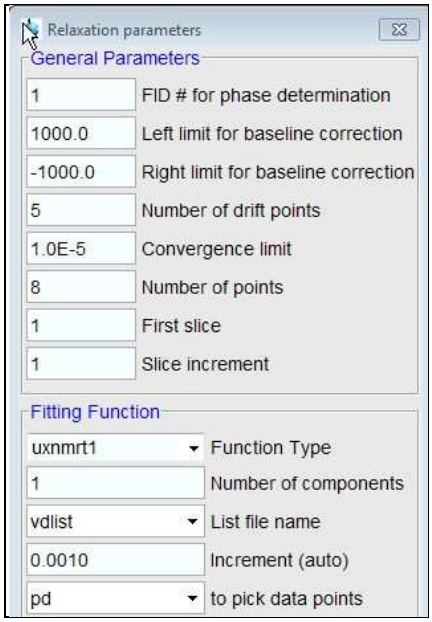
20) Click the **Relaxation Window** button, note any screen messages that may appear and close them. Select **Intensity** or **Area** in the plot window that appears. **Area** is often the better choice.

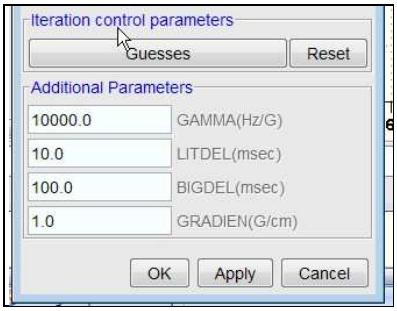


21) Click the **Fitting Function** button and note the comments about **intensity** or **are**a options in the screen display that appears and close it.



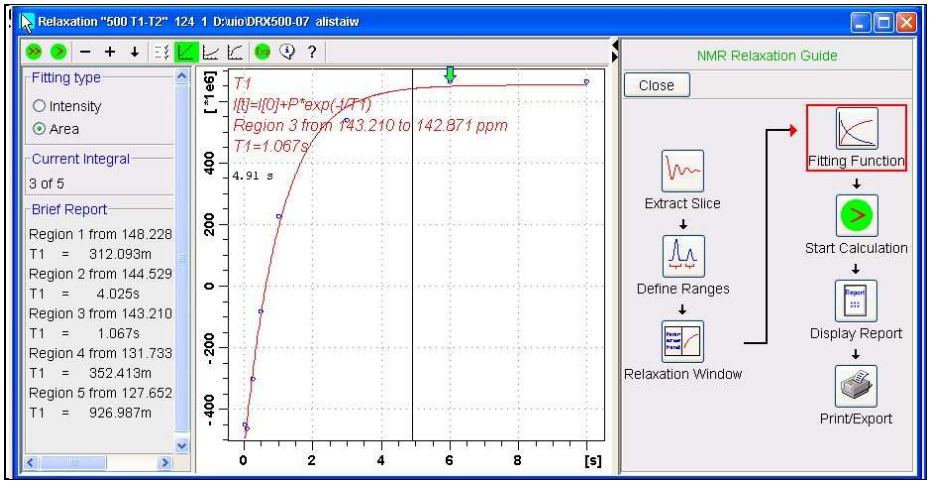
22) When the Fitting Function message screen is closed a panel with relaxation parameters (split into two screen captures below) will appear.





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

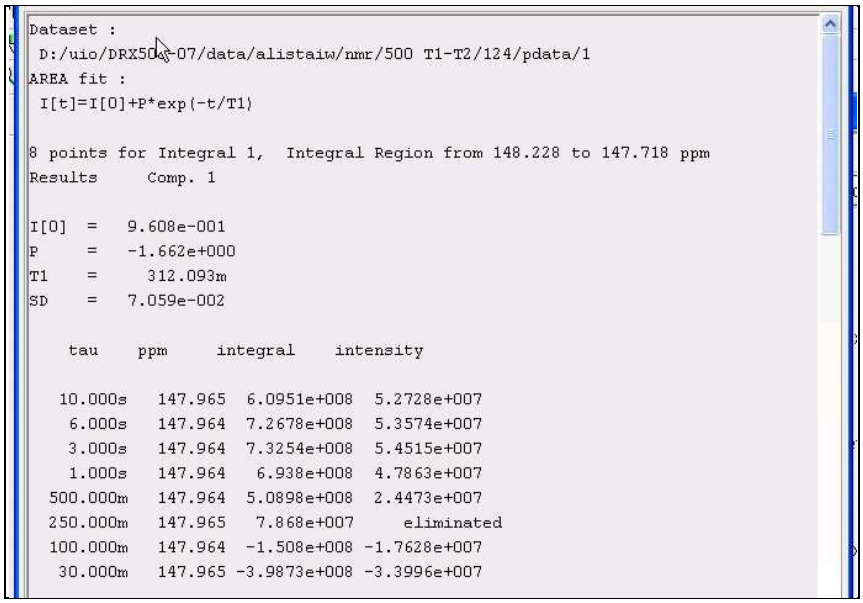
24) Click the **double red arrow** in the menu bar at the *top left* of the plot window and **NOT** the single red arrow button in the NMR Relaxation Guide menu below the Fitting Function button.



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 :
 D:/uio/DRX500-07/data/alistaiw/nmr/500 T1-T2/124/pdata/1
AREA fit :
 I[t]=I[0]+P*exp(-t/T1)

8 points for Integral 1,  Integral Region from 148.228 to 147.718 ppm
Results     Comp. 1

I[0]  =   9.608e-001
P     =   -1.662e+000
T1    =     312.093m
SD    =   7.059e-002

    tau    ppm     integral    intensity

   10.000s   147.965  6.0951e+008  5.2728e+007
    6.000s   147.964  7.2678e+008  5.3574e+007
    3.000s   147.964  7.3254e+008  5.4515e+007
    1.000s   147.964   6.938e+008  4.7863e+007
  500.000m   147.964  5.0898e+008  2.4473e+007
  250.000m   147.965   7.868e+007     eliminated
  100.000m   147.964  -1.508e+008 -1.7628e+007
   30.000m   147.965 -3.9873e+008 -3.3996e+007
```