



KJM 9250

**Carbon $T_2$  Spectra on the AVIII800HD Spectrometer**

Version 5.0

Topspin 3.5 Windows 7 AVIII HD 800



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# CarbonT<sub>2</sub> Spectra on the AVIII800HD

## 1.0 Introduction

An **awcarbont2** parameter set and linked **VCLIST** file from which a **VDLIST** file *must* be generated prior to processing a T<sub>2</sub> data set have been set up on the AVIII800HD spectrometer running under TS3.2 respectively.

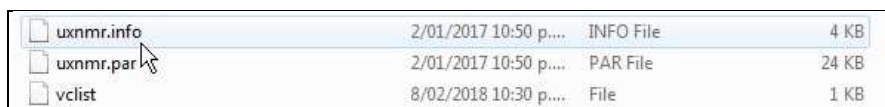
The parameter set have **D1 = 10 sec** and 8 x **VCLIST** values. The T<sub>2</sub> data set is acquired using the *smallest VCLIST value first. Prior to processing T<sub>2</sub> data sets variable constant (VC) values must be converted to time domain variable delay (VD) values using the **vctovd** command (see instructions15 and 16.)*

**D1** and the longest **VD** time derived from the largest **VC value** should be 3-5 times the longest T<sub>2</sub> in the sample compound. T<sub>2</sub> is always shorter than T<sub>1</sub>.

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

T<sub>2</sub> 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.

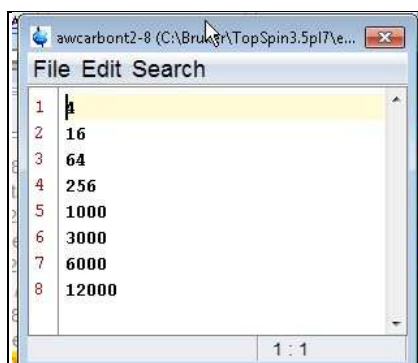


uxnmr.info	2/01/2017 10:50 p...	INFO File	4 KB
uxnmr.par	2/01/2017 10:50 p...	PAR File	24 KB
vclist	8/02/2018 10:30 p...	File	1 KB

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

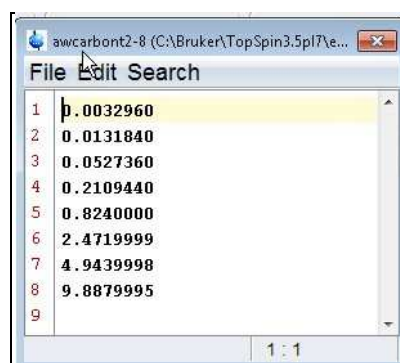
## 2.0 Experiment Set Up

- 1) Create an experiment and use the **rpar** command to read in the following parameter set: **awcarbont2** (d1 = 10 sec) + **getprosol**,  
pulse programme = **awcpmpgp**.  
Check **Digmod = Baseopt**
- 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**  
**D20 = 400 usec = 0.000400 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 **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) = 24 usec** and **D20 = 400 usec = 0.0004 sec** are shown below.



Line	VC Value
1	4
2	16
3	64
4	256
5	1000
6	3000
7	6000
8	12000

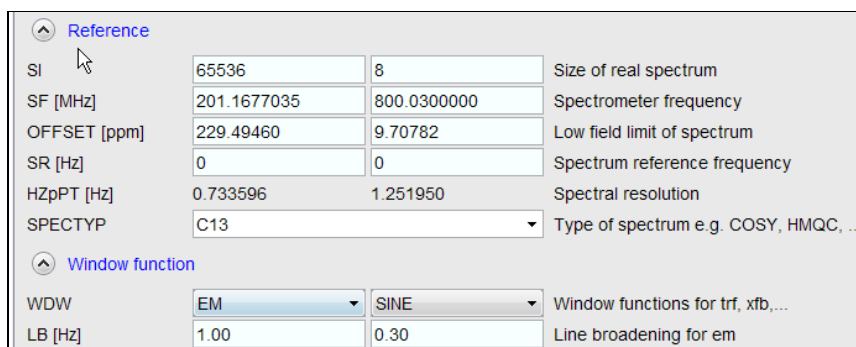
AWCARBONT2-8 VC file values



Line	VD Value (sec)
1	0.0032960
2	0.0131840
3	0.0527360
4	0.2109440
5	0.8240000
6	2.4719999
7	4.9439998
8	9.8879995
9	

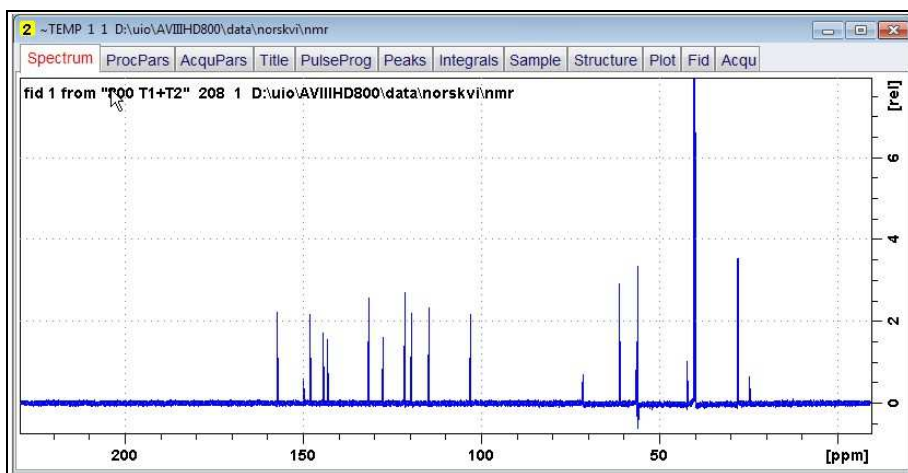
Calculated VD times (sec)

- 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.



Reference			
SI	65536	8	Size of real spectrum
SF [MHz]	201.1677035	800.0300000	Spectrometer frequency
OFFSET [ppm]	229.49460	9.70782	Low field limit of spectrum
SR [Hz]	0	0	Spectrum reference frequency
HZpPT [Hz]	0.733596	1.251950	Spectral resolution
SPECTYP	C13		
Window function			
WDW	EM	SINE	Window functions for trf, xfb, ...
LB [Hz]	1.00	0.30	Line broadening for em

- 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. The **PHC1** angle may be in the range -500 to -700 degrees.

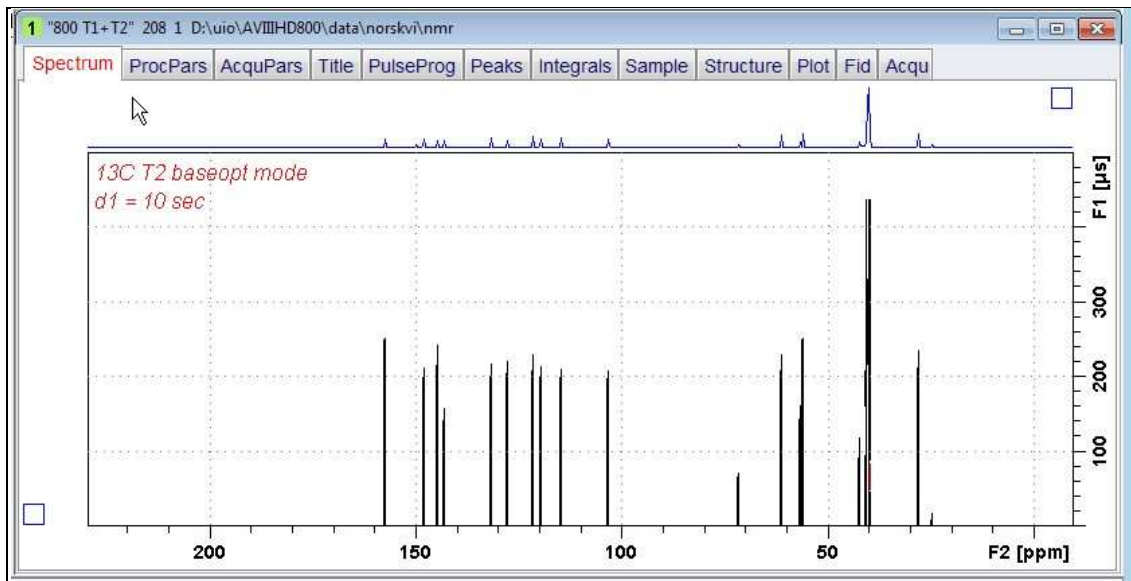
Phase correction	
PHC0 [degrees]	234.170
PHC1 [degrees]	-618.168
PH_mod	pk

- 11) Close the **TEMP** window and reload the T<sub>1</sub> 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. A large negative **PHC1** value will likely be required.

Phase correction			
PHC0 [degrees]	234.170	0	0th order correction for pk
PHC1 [degrees]	-618.168	0	1st order correction for pk
PH_mod	pk	mc	Phasing modes for trf, xfb, ...

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

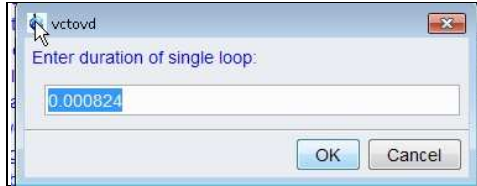
- 14) Provided phase constants have been correctly set up the transformed data set plot should resemble that shown below. Black = a positively phased signal. Signals will drop off to zero intensity as VD increases.



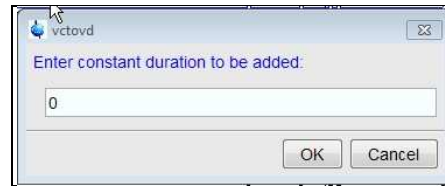
### 3.0 T<sub>1</sub> Data Set Processing

15) Prior to processing a T<sub>2</sub> 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.

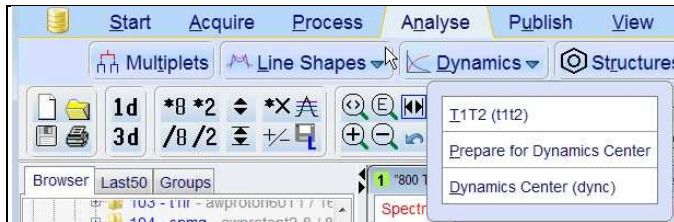


Single VC loop value =  $p2 + 2 \times d20$



Not used. .

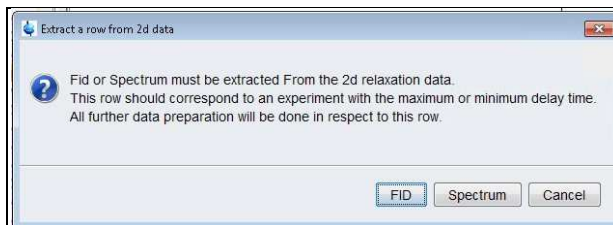
17) Click Topspin's **Analyse** menu tab then its **Dynamics** sub-menu tab and click its **T1T2** tab.



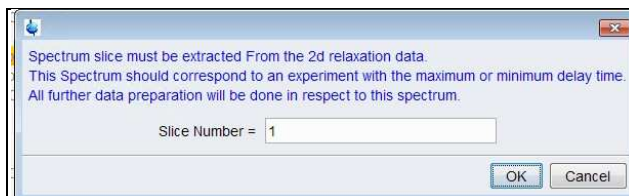
18) This will open the T1/T2 processing menu bar shown below.



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

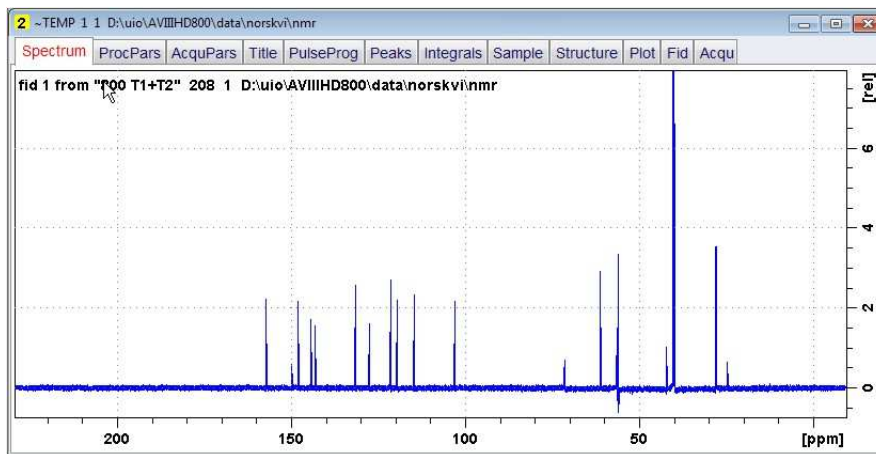


19) The screen panel shown below will appear.

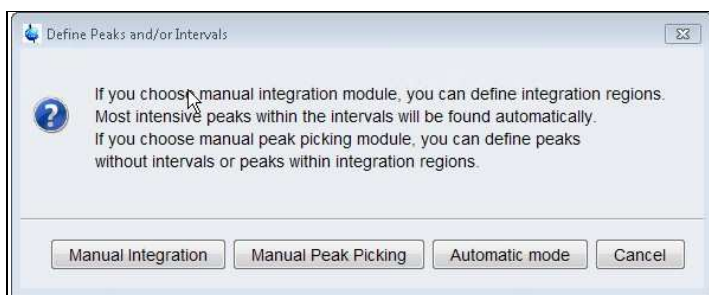
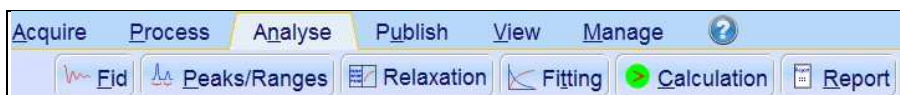


Enter **1** in the **Slice Number** cell and click the **OK** button. This will display the transformed spectrum ex the first (longest) **VDLIST** value = the one that was phased via the **rser 1** routine in steps 8 and 9.

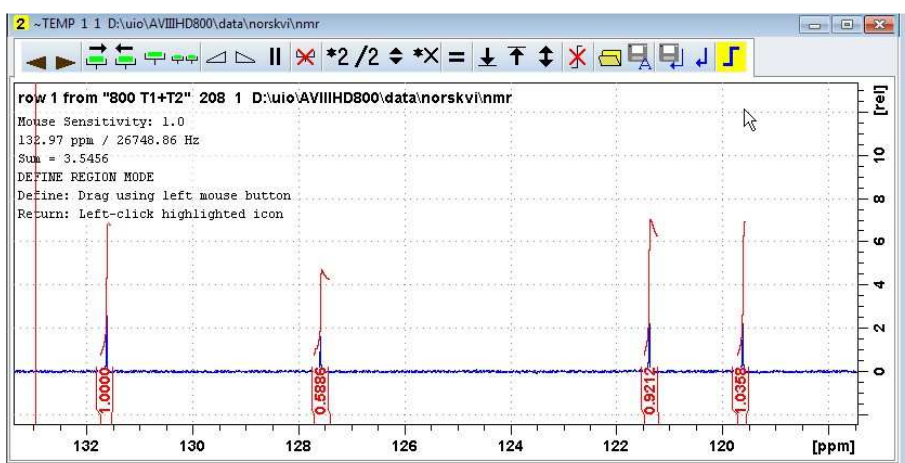
(...next page)



20) 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 .

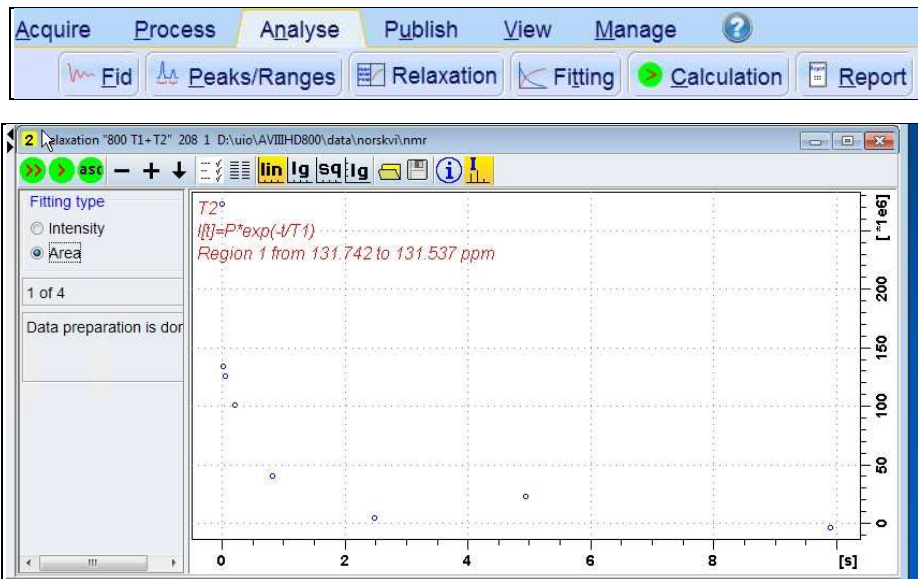




- 21) Click the "Save Region As" icon button (= the floppy disk icon with A below it) and then its **Export Regions to Relaxation Module and .ret.** tab.

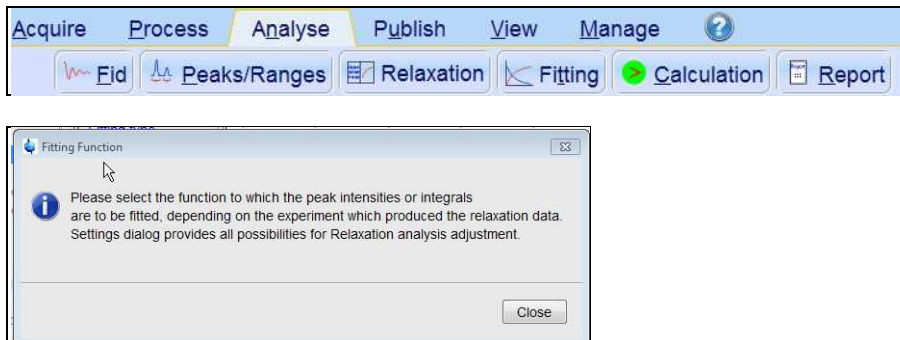


- 22) Click the **Relaxation** menu tab button, note any screen messages that may appear and close them. Select **Intensity** or **Area** in the plot window that appears. **Area** is invariably the better choice (next page).



Note: The T1 equation appears in error in this display.

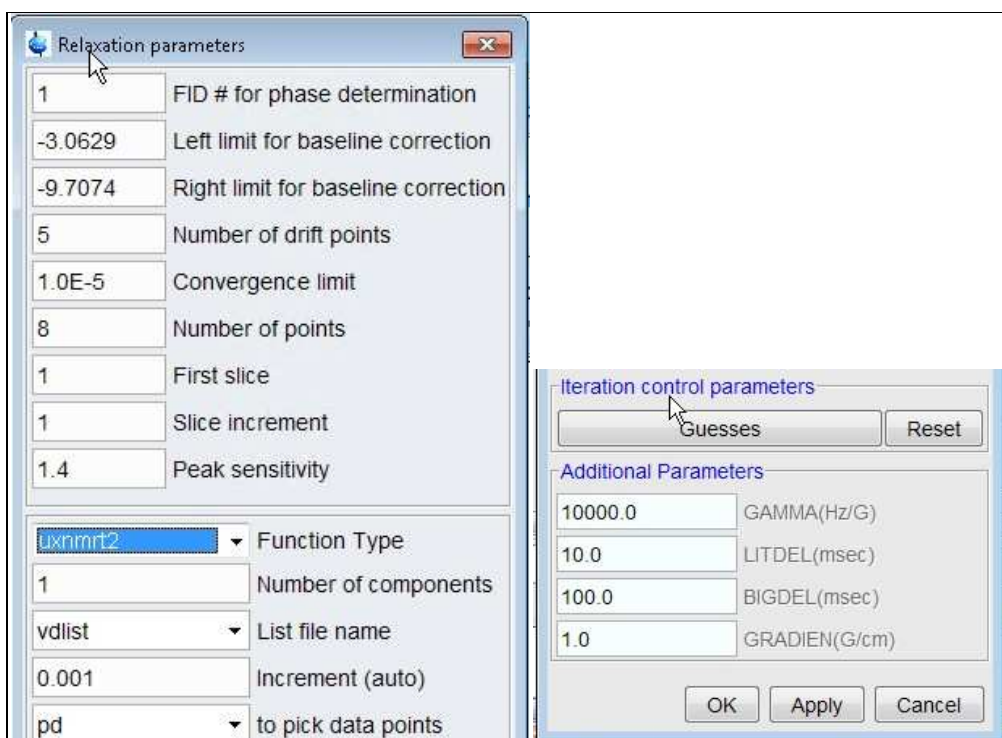
- 23) Click the **Fitting** menu bar tab and note the comments about **intensity** or **area** options in the screen display that appears and close it



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

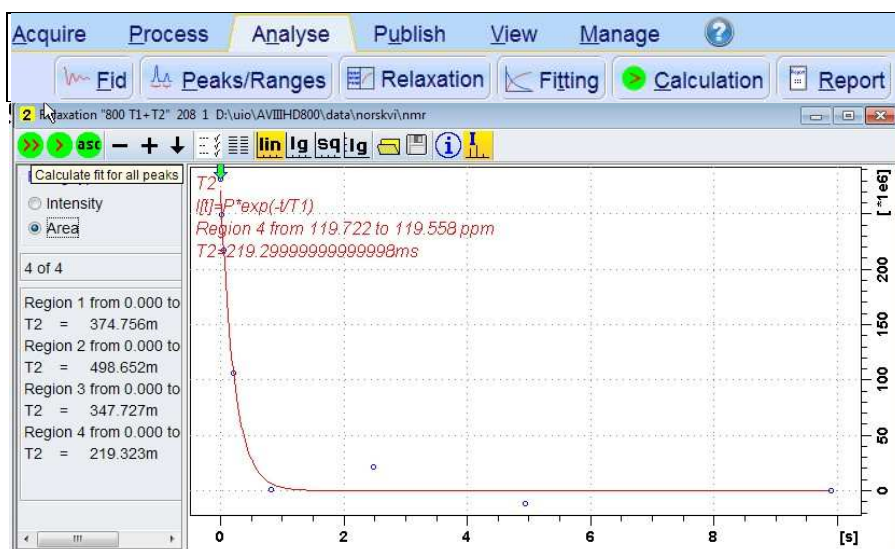
(...next page\_





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

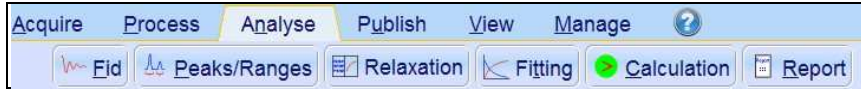
26) Click the **double red arrow** in the T2 plot's menu bar and **NOT** the **Calculation** tab in the upper menu bar.



The plot's upper title line correctly has T<sub>2</sub>, but the T<sub>1</sub> equation incorrectly appears below it. This is a long standing Topspin bug.

27) The + and - buttons in the upper menu bar can be used to advance (or reverse) the individual T<sub>1</sub> 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<sub>1</sub> results and update their plots.
- 29) 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 :
2 D:/uio/AVIIHD800/data/norskvi/nmr/800 T1+T2/208/pdata/1
3 AREA fit :
4 I[t]= P*exp(-t/T2)
5
6 8 points for Integral 1, Integral Region from 0.000 to 0.000 ppm
7 Results Comp. 1
8
9 P = 6.630e-001
10 T2 = 374.756m
11 SD = 1.438e-001
12
13 tau ppm integral intensity
14
15 3.296m 131.615 2.7275e+008 5.933e+007
16 13.184m 131.616 1.3444e+008 3.1e+007
17 52.736m 131.614 1.2545e+008 3.4431e+007
18 210.944m 131.615 1.0141e+008 2.2007e+007
19 824.000m 131.615 4.0072e+007 4.2391e+006
20 2.472s 131.618 4.6224e+006 eliminated
21 4.944s 131.618 2.2506e+007 eliminated
22 9.888s 131.618 -3.6614e+006 eliminated

```