



KJM 9250

CarbonT₂ Spectra on the AVIII800HD Spectrometer

Version 1.0

Topspin 3.5 Windows 7 AVIII HD 800



© Professor Emeritus Alistair Lawrence Wilkins,
University of Waikato, New Zealand.
April 2018

CarbonT₂ 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 T₂ 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 T₂ data set is acquired using the smallest VCLIST value first. Prior to processing T₂ 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 T₂ in the sample compound. T₂ is always shorter than T₁.

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

T₂ 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.

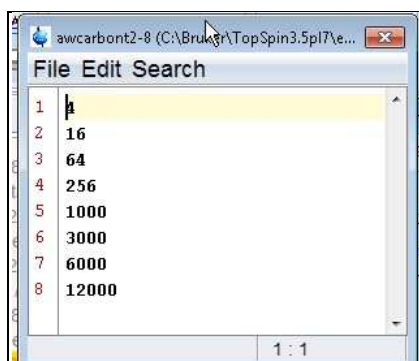
If required the VCLIST file that is present in the experiment's top level folder can be opened with **WordPad** and the **VC** values in it can be viewed and used to recreate the original named **VCLIST** file from which the **VDLIST** file can be created using the **vctovd** command.

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.

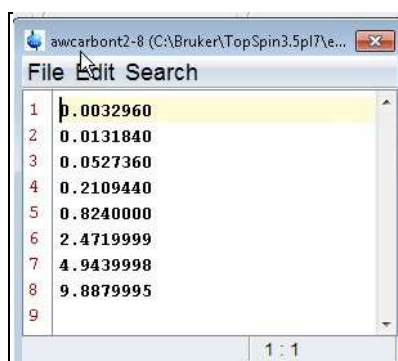
2.0 Experiment Set Up

- 1) Create an experiment and use the **rpar** command to read in the following parameter set: **awcarbont2** (d1 = 10 sec) + **getprosol**,
pulse programme = **awcpmgrp**.
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.



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

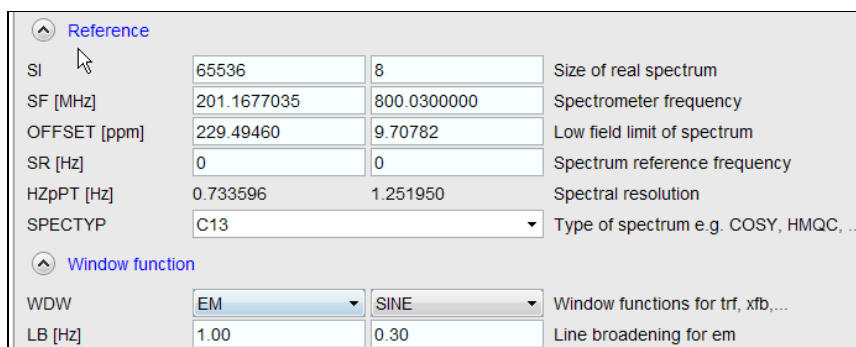
AWCARBONT2-8 VC file values



	VD (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

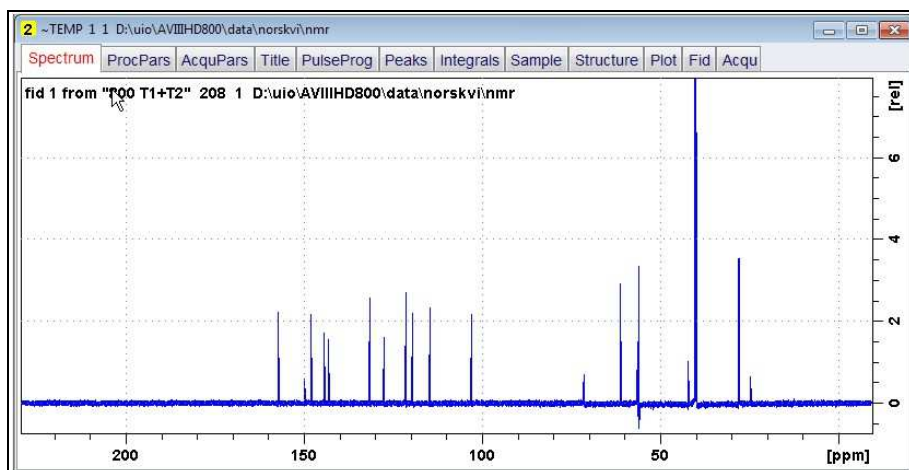
Calculated VD times (sec)

- 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		Type of spectrum e.g. COSY, HMQC, ...
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

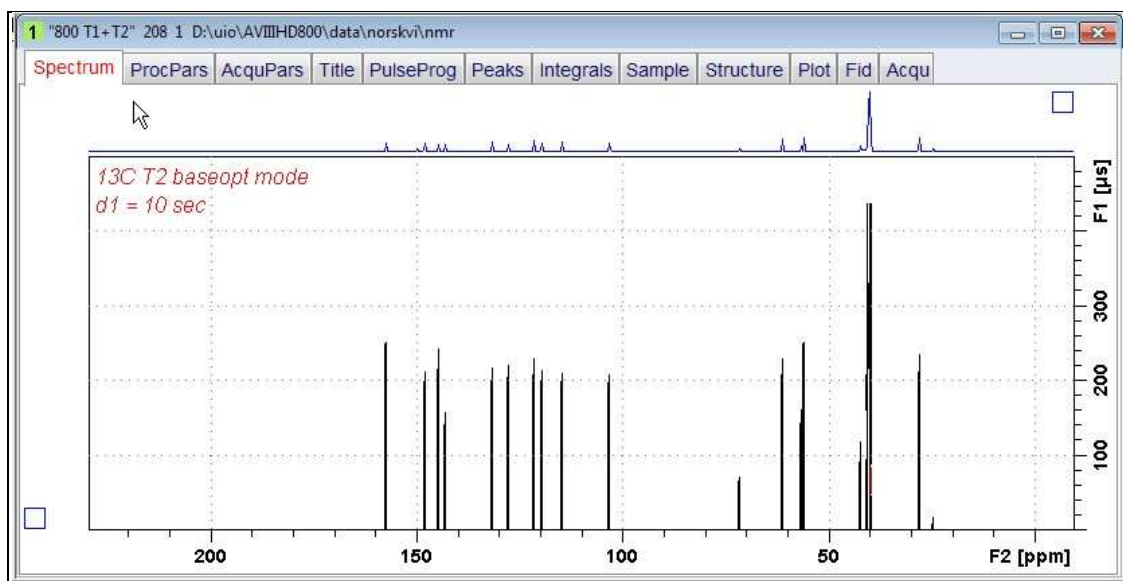
- 11) Close the **TEMP** window and reload the T₁ data set file.
- 12) Type **edp** (enter) and enter the phase constants noted in step 10 above into the **F2 PHC0** and **PHC1** cells and check **PH_MOD** = **pk**. **F1** cell info is not used. A large negative **PHC1** value will likely be required.

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.

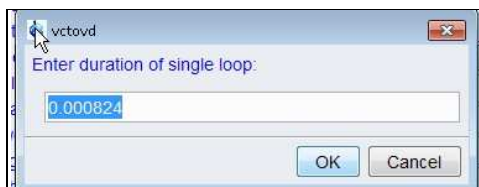
- 14) Provided phase constants have been correctly set up the transformed data set plot should resemble that shown below. Black = a positively phased signal. Signals will drop off to zero intensity as VD increases.



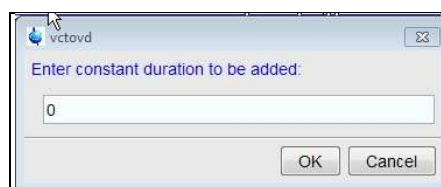
3.0 T₁ Data Set Processing

15) Prior to processing a T₂ 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.

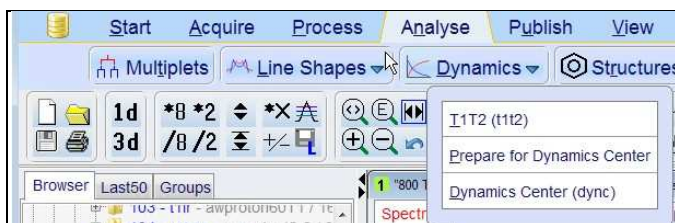


Single VC loop value = $p2 + 2 \times d20$

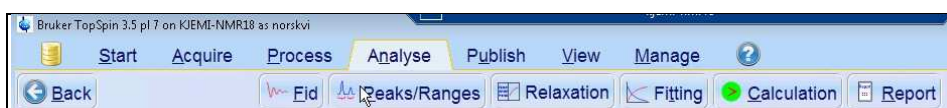


Not used. .

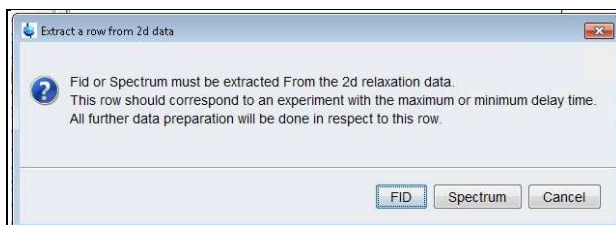
17) Click Topspin's **Analyse** menu tab then its **Dynamics** sub-menu tab and click its **T1T2** tab.



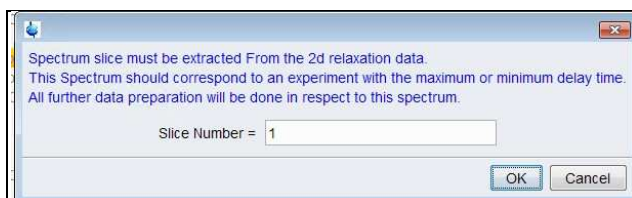
18) This will open the T1/T2 processing menu bar shown below.



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

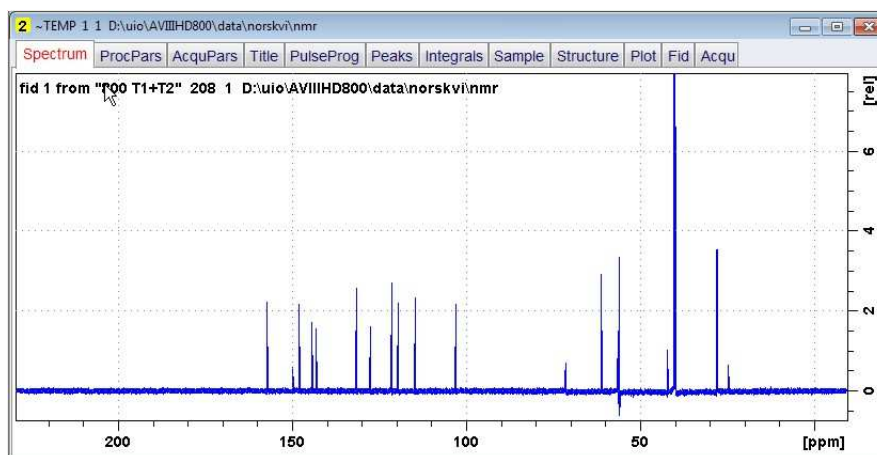


19) The screen panel shown below will appear.

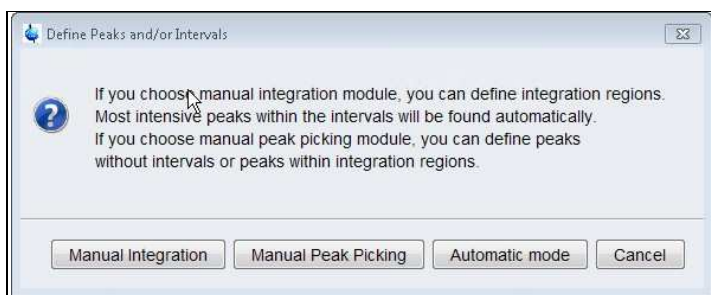
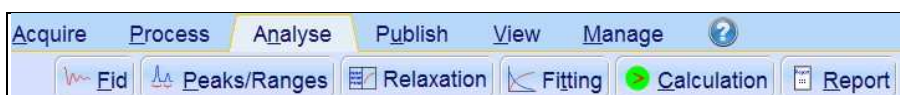


Enter **1** in the **Slice Number** cell and click the **OK** button. This will display the transformed spectrum ex the first (longest) **VDLIST** value = the one that was phased via the **rser 1** routine in steps 8 and 9.

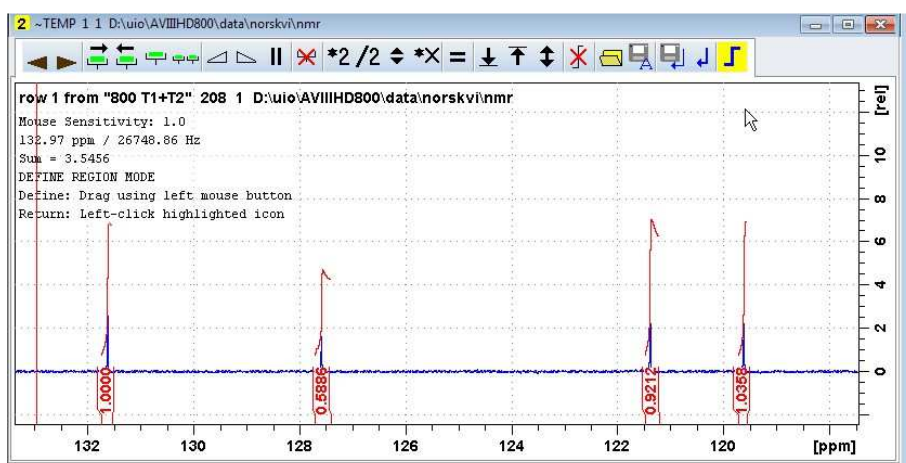
(....next page)



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



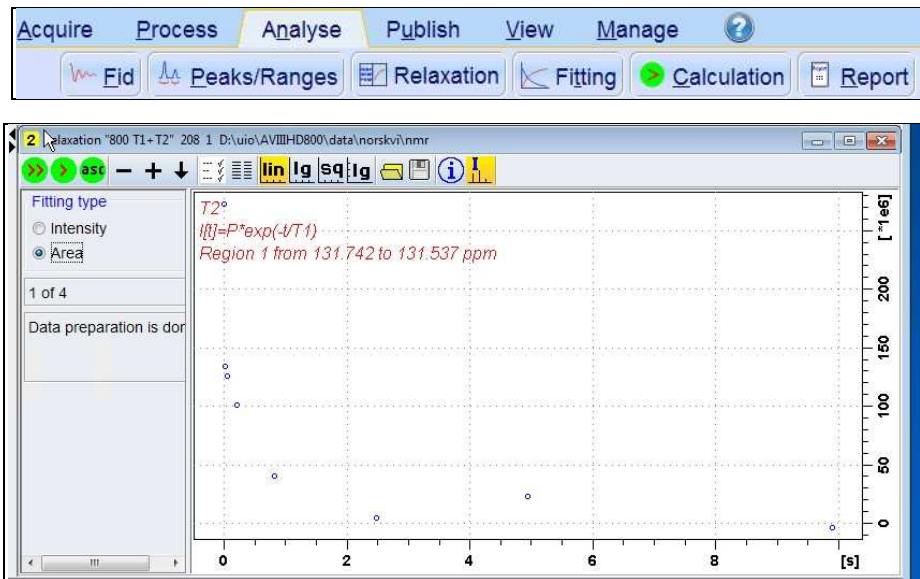
Select the **Manual integration** tab, close any screen messages that appear, and integrate selected peaks in the usual way .



- 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.

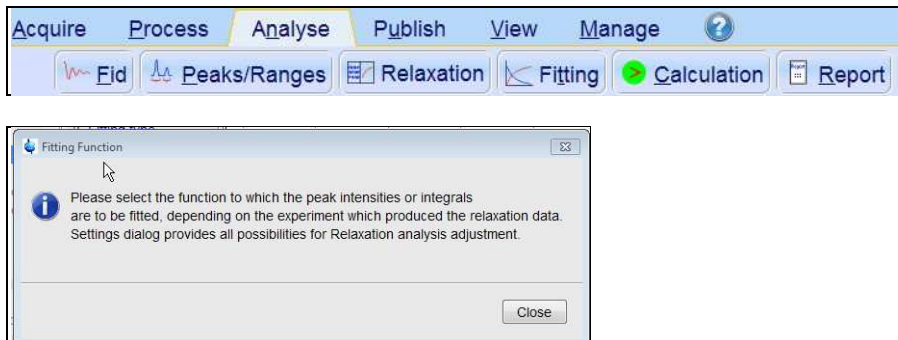


- 22) Click the **Relaxation** menu tab button, note any screen messages that may appear and close them. Select **Intensity** or **Area** in the plot window that appears. **Area** is invariably the better choice (next page).



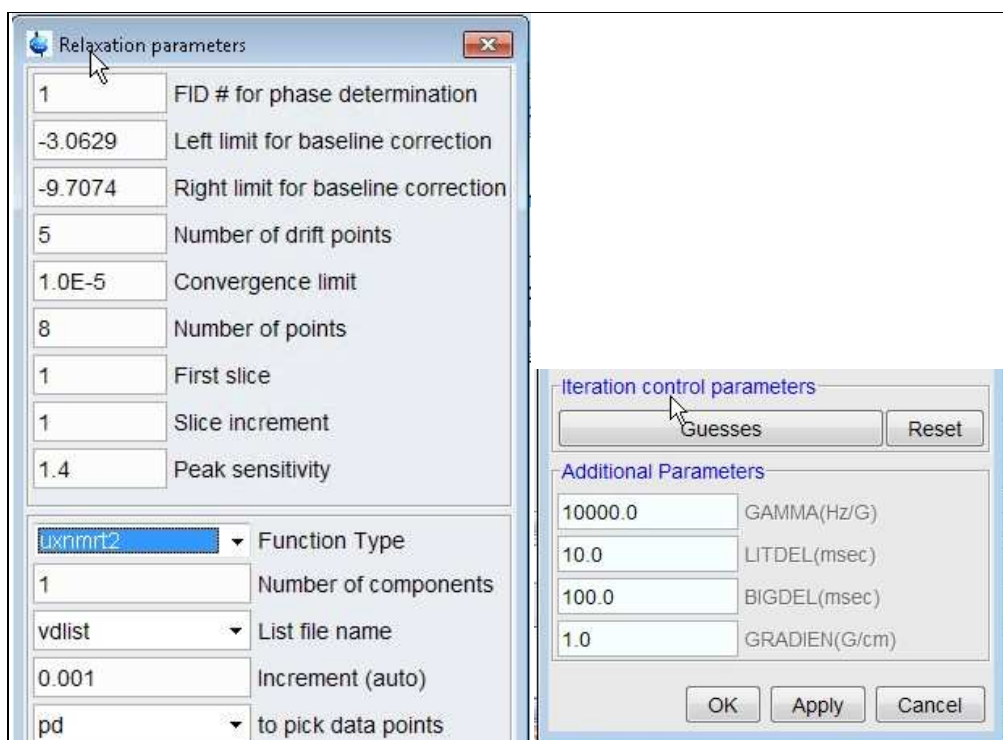
Note: The T1 equation appears in error in this display.

- 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



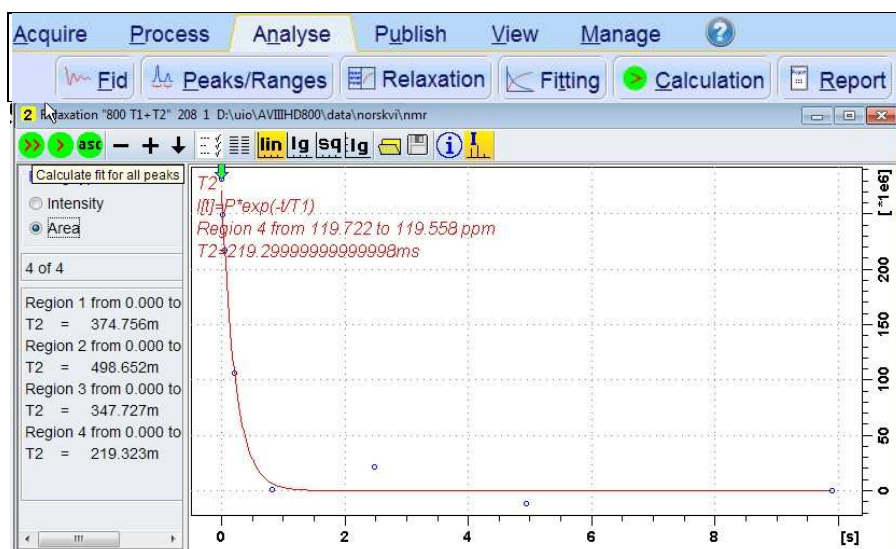
- 24) When the **Fitting** function message screen is closed a panel with **Relaxation parameters** (split into two screen captures below) will appear.

(...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.

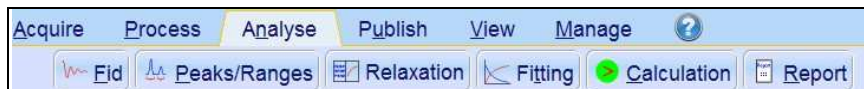
- 26) Click the **double red arrow** in the T2 plot's menu bar and **NOT** the **Calculation** tab in the upper menu bar.



The plot's upper title line correctly has T₂, but the T₁ 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 T₁ 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_1 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

1

2

3

4

5

6

7

8

9

10

11

12

13

14

15

16

17

18

19

20

21

22

Dataset :

D:\uio\AVI11HD800\data\norskvi\nmr\800 T1+T2/208/pdata/1

AREA fit :

$I[t] = P \cdot \exp(-t/T_2)$

8 points for Integral 1, Integral Region from 0.000 to 0.000 ppm

Results Comp. 1

P = 6.630e-001

T2 = 374.756m

SD = 1.438e-001

tau	ppm	integral	intensity
3.296m	131.615	2.7275e+008	5.933e+007
13.184m	131.616	1.3444e+008	3.1e+007
52.736m	131.614	1.2545e+008	3.4431e+007
210.944m	131.615	1.0141e+008	2.2007e+007
824.000m	131.615	4.0072e+007	4.2391e+006
2.472s	131.618	4.6224e+006	eliminated
4.944s	131.618	2.2506e+007	eliminated
9.888s	131.618	-3.6614e+006	eliminated