

KJM 9250

CarbonT₁ Spectra on the DRX500 Spectrometer

Version 5.0

Topspin 1.3 Windows XP DRX 500



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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 <u>longest delay (10 sec) the first used VDLIST</u> 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.

T₁ 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.

🗋 uxnmr.info	2/01/2017 10:50 p	INFO File	4 KB
uxnmr.par	2/01/2017 10:50 p	PAR File	24 KB
📄 vdlist	10/01/2018 8:49 a	File	1 KB

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

- 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.

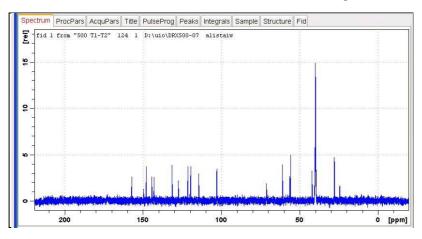
(i	AWPROTONT1-8 (C:\Bruker\TopSpin3.0\exp\stan\nmr\lists\vd)
Pvi	e Edit Search
1	10
2	6
3	3
4	1
5	0.5
6	0.25
7	0.10
8	0.03

- 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.

si 🖌	65536	8	Size of real spectrum
SF [MHz]	125.7577890	500.1300000	Spectrometer frequency
OFFSET [ppm]	219.39250	4.99974	Low field limit of spectrum
SR [Hz]	0	0	Spectrum reference frequency
HZpPT [Hz]	0.458222	125.000000	Spectral resolution
SPECTYP	UNDEFINED		 Type of spectrum e.g. COSY, HI
Window function	tion		
WDW	EM	▼]SINE	Window functions for trf, xfb,
LB [Hz]	2.00	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. The **multiabsn** command with n = 30-40 can be used to straighten the baseline.



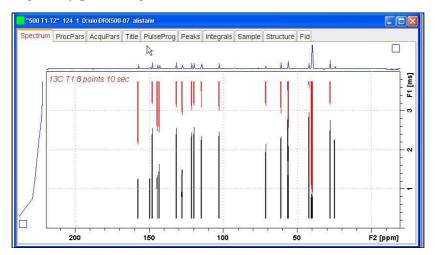
10) Type edp (enter) and note the phase constants for this spectrum.



- 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.

Phase correction					
PHC0 (degree) =	252.218		0.000		Oth order correction for pk
PHC1 [degree] =	12.970		0.000		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, red = a negatively phased signal.

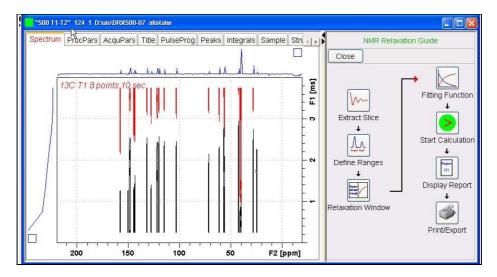


3.0 T₁ Data Set Processing

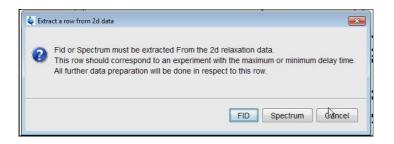
15) Open Topspin's Analysis menu and click its T1/T2 Relaxation tab.

💐 Bruker TOPSPIN 1.3 on KJEMI-NMR07 as norsky	vi 🔍		kjen	ni-nmr07
File Edit View Spectrometer Proces	ssing	Analysis	Options Window H	lelp
□ ③ □ ♀ ● ► ► Id 3d 人 ふ ⊥ *2 /2 *8 /8 ≑ *X 至 夫 *2 見 Ⅲ 臣 ○ Browser PFolio Alias		Peak Pi	ibration [cal] cking [pp] on [int]	💿 V
e 🔄 alistaiw	Spec	Multiple	Spectrum Display	'rog P
⊖ <u>3</u> 500 T1-T2		Deconv	olution	
2		T1/T2 F	elaxation	mula
₩ <u>3</u> ₩ <u>11</u>		Simulate	Fid	111

This will open up the screen display shown below.



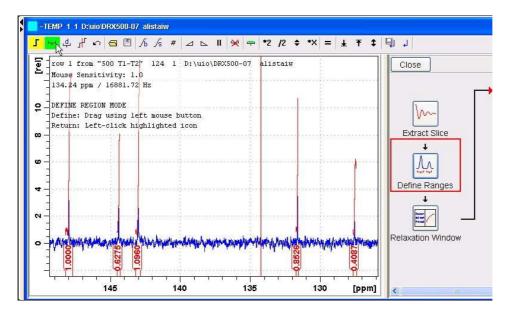
16) Click the **Extract Slice** button and then the click the **Spectrum** button in the panel that appears.



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.

		relaxation data.		
This Spectrum should con			kimum or mir	nimum delav time.
All further data preparatio				acia) anio
Slice	Number =			
Sille				

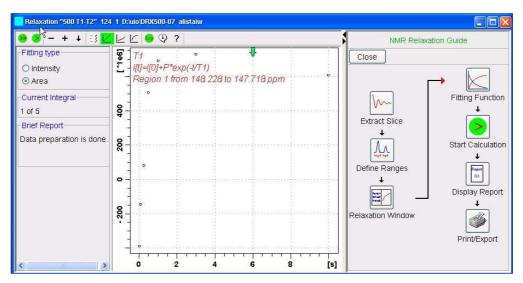
18) Expand the spectrum that appears in the usual way and integrate selected peaks in it. Integrals should start and terminate as close as possible to the edge of a peak.



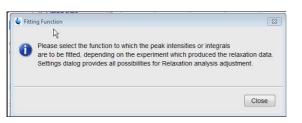
19) Click the "Save Region As" icon button (= 7th from the left in TS1.3's upper menu bar = the one with the floppy disk icon) and then its Export Regions to Relaxation Module and .ret. tab.

· · · · · · · · · · · · · · · · · · ·	Ⅱ 👾 🖛 *2 /2 💠 *X = ± ∓ ‡ 🗐 ↓
5 row 1 from "500 T1-T2" 124 1 D:\uio\	Save Regions To 'intrng'
Mouse Sensitivity: 1.0 133.86 ppm / 16833.61 Hz	Save Regions To 'reg'
• DEFINE REGION MODE	Export Regions To Relaxation Module and .ret.

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.



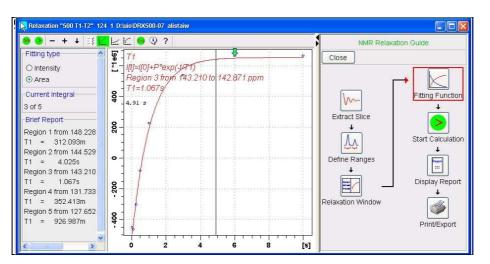
21) Click the Fitting Function button and note the comments about intensity or area options in the screen display that appears and close it.



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

	arameters		
1	FID # for phase determination		
1000.0	Left limit for baseline correction		
-1000.0	Right limit for baseline correction		
5	Number of drift points		
1.0E-5	Convergence limit		
B	Number of points		
	and the second		
1	First slice	-Iteration contr	ol parameters
1	First slice Slice increment		ol parameters Guesses Reset
1	Slice increment		Guesses
1 Fitting Fun	Slice increment		Guesses
1 Fitting Fun uxnmrt1	Slice increment	Additional Par	Guesses Reset
1 Fitting Fun	Slice increment	Additional Par 10000.0	GAMMA(Hz/G)

- 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 <u>top left</u> of the plot window and <u>NOT</u> 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 T₁ 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 T₁ results and update their plots.
- **27**) The NMR Relaxation Guide has buttons which can be used to display and/or print T₁ results. A sample report for one signal is shown below.

```
Dataset :
  D:/uio/DRX50 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
        = -1.662e+000
P
т1
        =
              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
```