KJM 9250

CarbonT_2 Spectra on the AVIII800HD Spectrometer

Version 5.0

Topspin 3.5 Windows 7 AVIII HD 800

April 2018

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1.0 Introduction

An **awcarbont2** parameter set and linked **VCLIST** file from which a **VDLIST** file *must* be generated prior to processing a T_2 data set have been set up on the AVIII800HD spectrometer running under TS3.2 respectively.

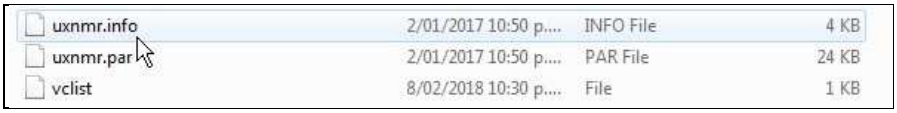
The parameter set have **D1 = 10 sec** and 8 x **VCLIST** values. The T_2 data set is acquired using the *smallest VCLIST value first. Prior to processing T_2 data sets variable constant (VC) values must be converted to time domain variable delay (VD) values using the **vctovd** command (see instructions15 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.

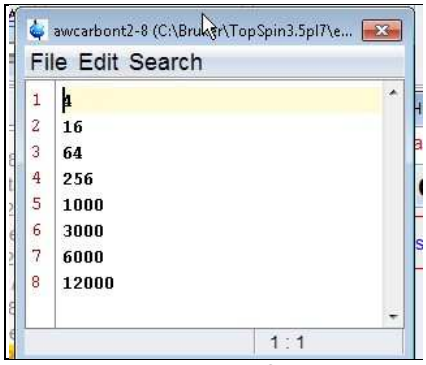
2.0 Experiment Set Up

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

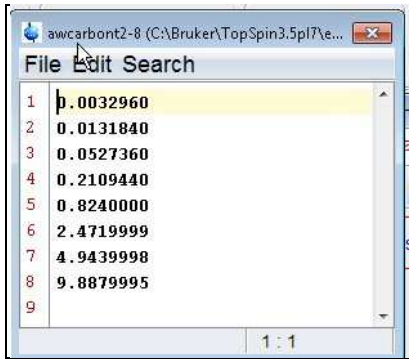
2) Review default settings. These settings can be adjusted if required.
TD(F2) = 64K or 32K,**TD(F1) = 8**
SW = 240 ppm, O1P = 110 ppm.
D1 = 10 sec
D20 = 400 usec = 0.000400 sec
NS = a **multiple of 2, 4 or 8, DS = 0, 2 or 4.**

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.



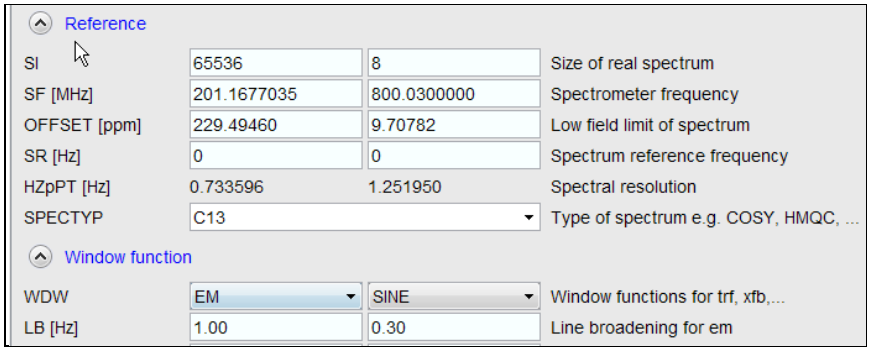
AWCARBONT2-8 VC file values



Calculated VD times (sec)

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

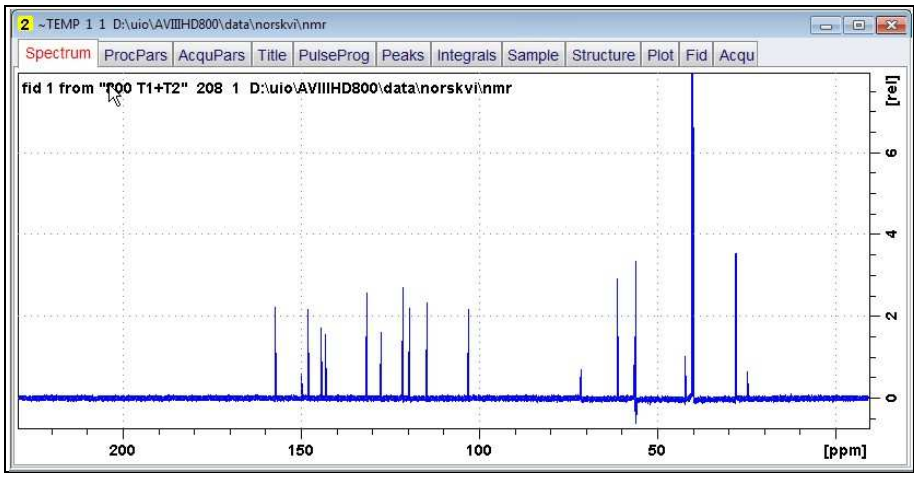
6) Type **edp** (enter) and check that **SI(F2) = 32 or 64K**, **SI(F1) = 8, WDW(F2) = EM, LB (F2) = 1.0-2.0 Hz** or other value of your choice.



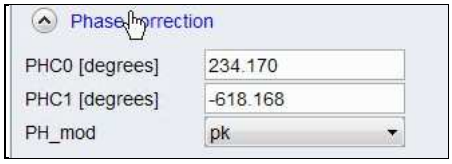
7) Start the acquisition using the **ZG** command.

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.

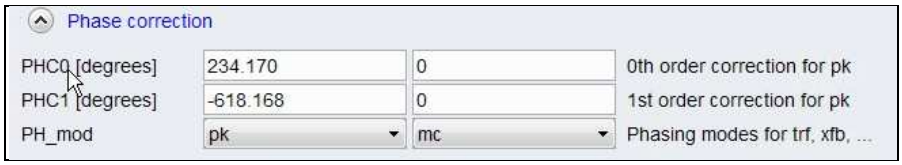


10) Type **edp** (enter) and note the phase constants for this spectrum. The **PHC1** angle may be in the range -500 to -700 degrees.



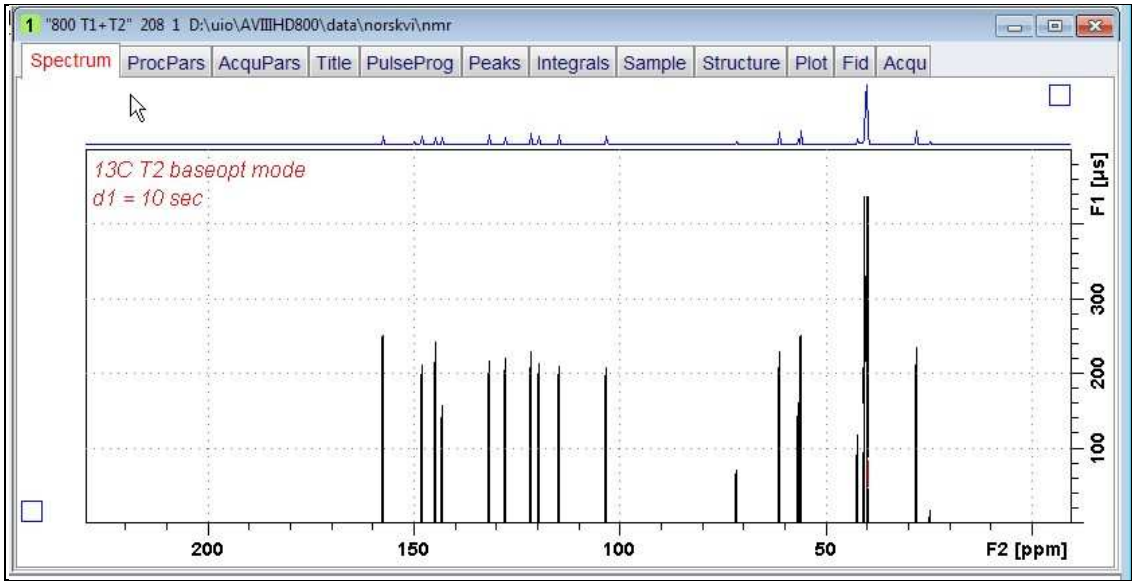
11) Close the **TEMP** window and reload the T_1 data set file.

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. A large negative **PHC1** value will likely be required.



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

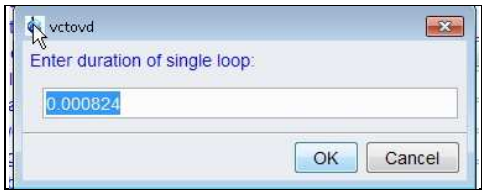
14) Provided phase constants have been correctly set up the transformed data set plot should resemble that shown below. Black = a positively phased signal. Signals will drop off to zero intensity as VD increases.



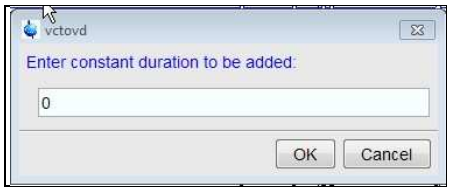
3.0 T_1 Data Set Processing

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.

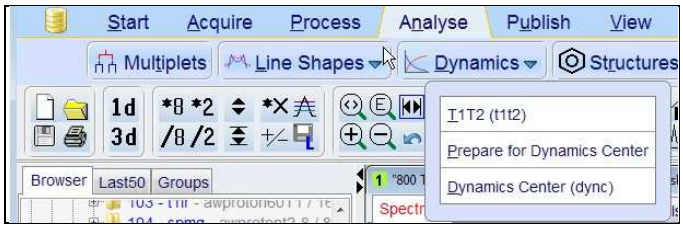


Single VC loop value = p2 + 2 x d20



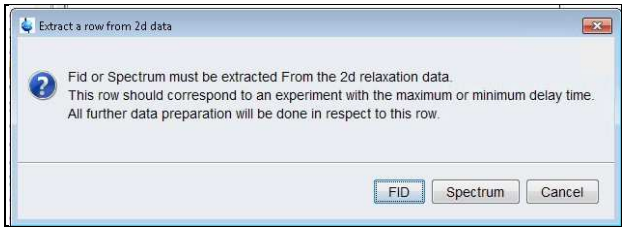
Not used. .

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

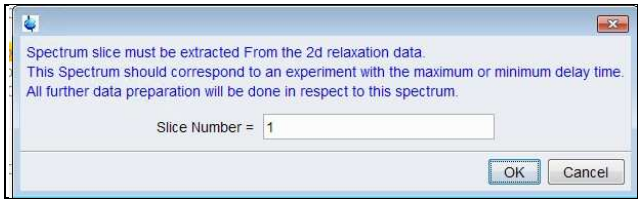


18) This will open the T1/T2 processing menu bar shown below.

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.

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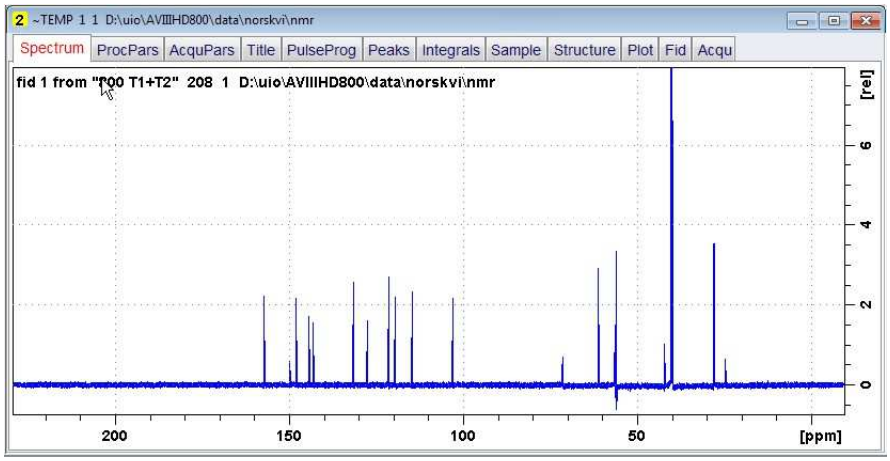
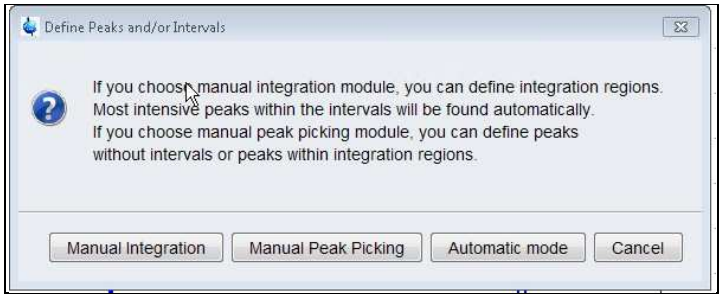
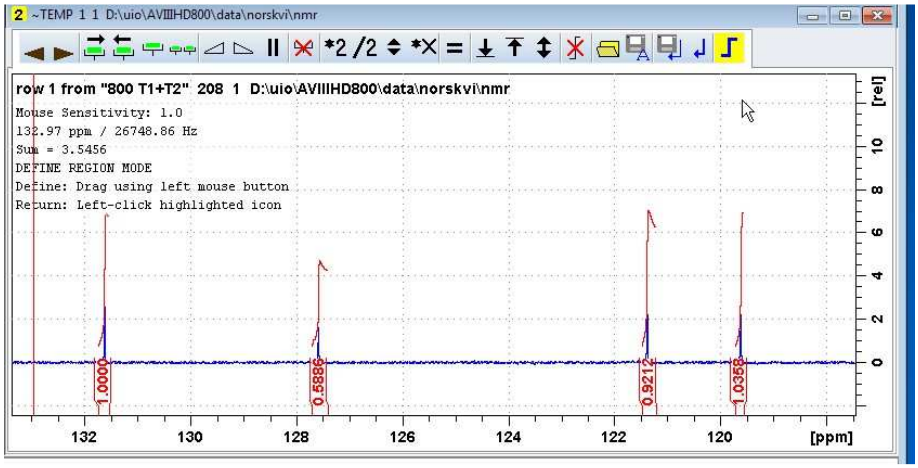


20) Expand the spectrum that appears in the usual way and click the **Peaks/Ranges** menu bar tab.

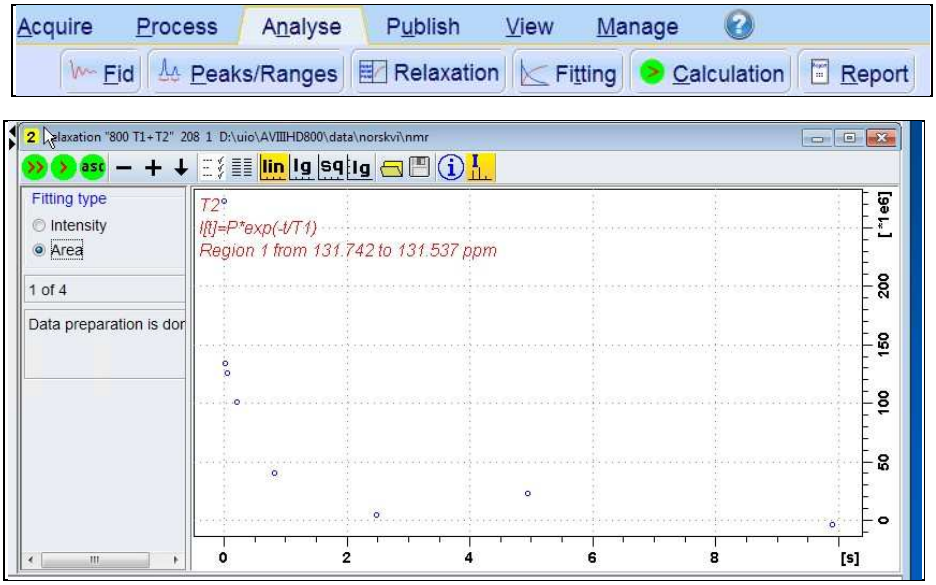


Select the **Manual integration** tab, close any screen messages that appear, and integrate selected peaks in the usual way .



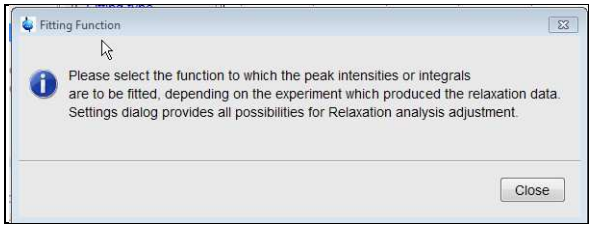
21) Click the "**Save Region As**" icon button (= *the floppy disk icon with A below it*) and then its **Export Regions to Relaxation Module and .ret**. tab.

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 **a**nd 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.

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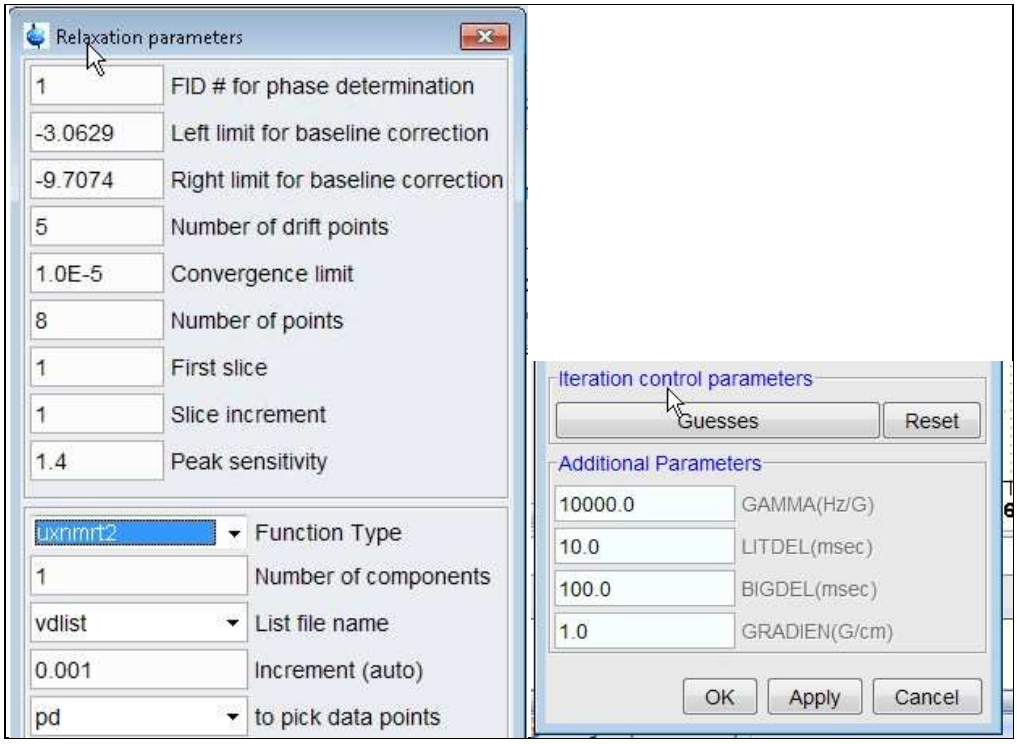
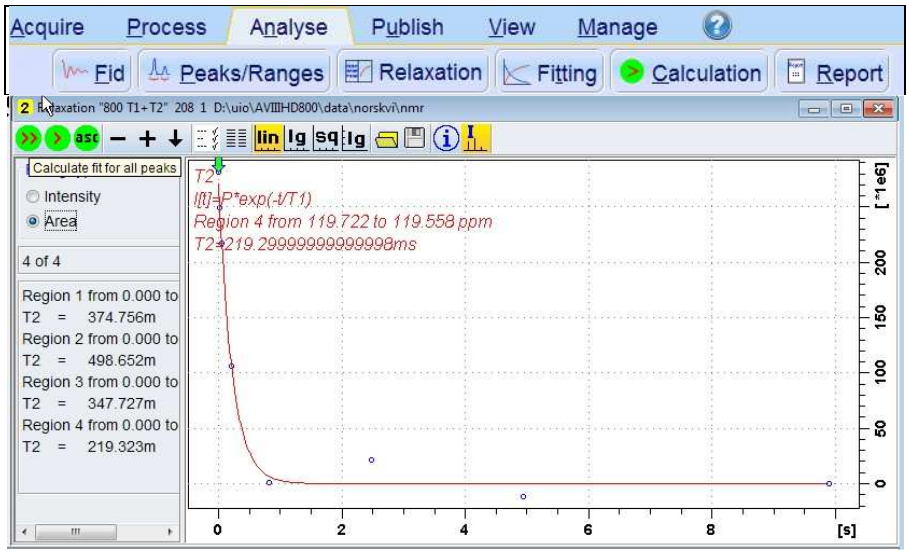


25) Check **Function Type = uxnmrt2** *(important!)* and **List file name = vdlist**.
Other cells/values can be left as they are (= default settings).
Click **Apply** and **OK** and close the window.

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



. *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.*

27) The + and - buttons in the upper menu bar can be used to advance (or reverse) the individual T_1 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