University of Puerto Rico
NMR LAB
STANDARD OPERATING PROCEDURE: NMR-02

TITLE: **Proton T_2 Measurement**		
Original Issue: 21 MAY 2006	Revision Date: 21 AUG 2006	**Page 1 of 5**

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Introduction

Tranverse relaxation (T_2) is the mechanism by which the excited magnetization vector (convenitonally shown in the *x-y* plane) decays. This is always at least slightly faster than longitudinal relaxation. The magnitude of the magnetic moment in the *x-y* plane decays according to

$$M = M_0 \exp\left(\frac{t}{T_2}\right)$$

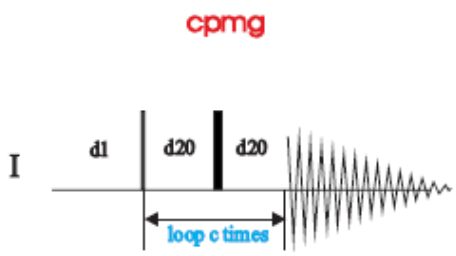
The CPMG (Carr-Purcell-Meiboom-Gill) (T_2) experiment yields a signal of intensity

$$I_0 \exp\left(\frac{-t}{T_2}\right)$$

where t is the total evolution time ($4n\Delta$). The value of Δ (d20) in the pulse sequence should be much shorter than the reciprocal coupling constant 1/J but long enough that the sample should not heat up significantly. A Δ (d20) of 10 ms is usually appropriate. The experiment is repeated many times with different values of τ and the resulting intensities used to find the value of T_2. This experiment works best for singlets. If the sample is very concentrated then the relaxation time will appear shorter than it really is due to saturation. In such a case, off-tune the probe, recalibrate the pulse widths and repeat the experiment.

We assume that the main program (TopSpin) to operate the instrument and the lock display (**[lockdisp]**↵) were activated previously. It also assumed that you previously ran a T1 experiment for the same sample.

Figure 1: *Carr-Purcell-Meiboom-Gill Pulse Sequence*



Procedure

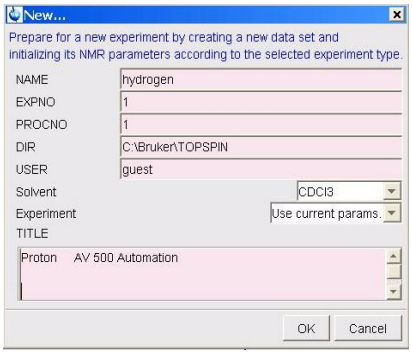
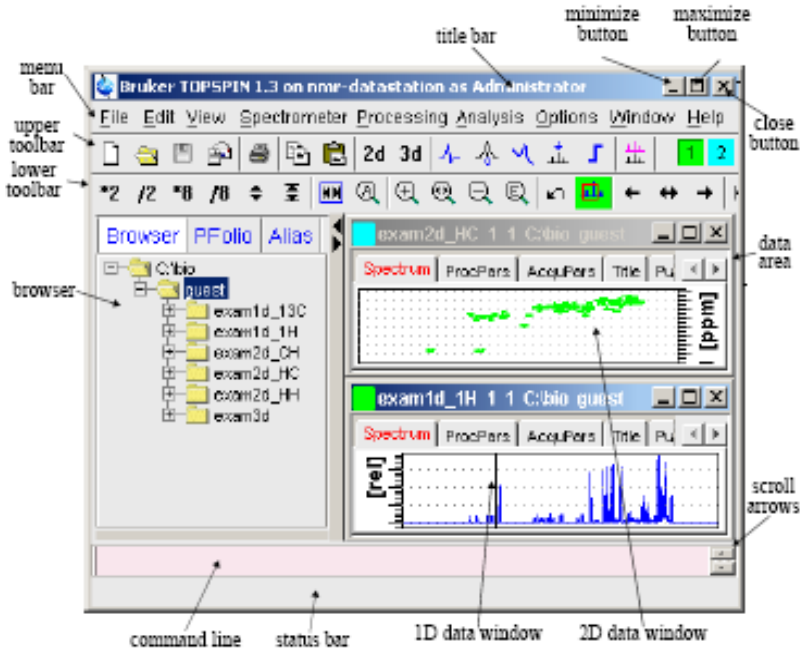
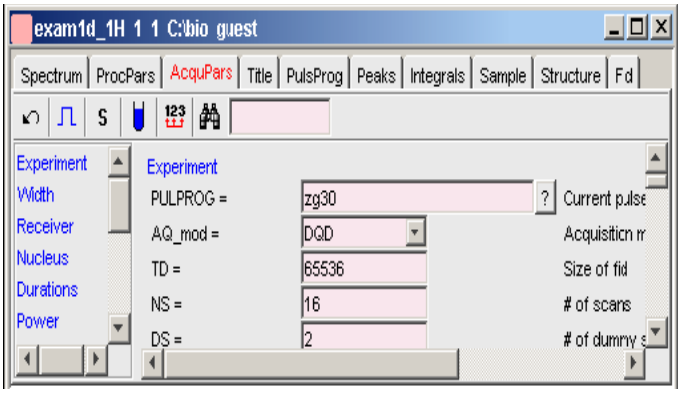
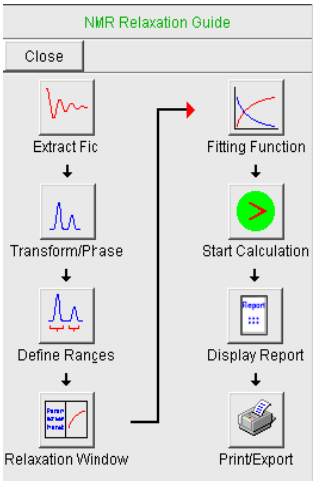
Step(s) and command(s)	Comment(s)
1. **[edc]** ↵  **Solvent:** the sample solvent **Experiment:** PROTON **Title**: T2 experiment of John Doe	Create the data set to record a ^{1}H reference spectrum. ↵ : means press enter in the keyboard. **Important:** DIR= **/opt/topspin** USER= **NMR** Experiment= PROTON
In the command line (see Figure 2) type 2. **[ej]** ↵ (to take out the previous sample) 3. introduce sample in spinner and measure sample depth 4. insert sample in magnet 5. **[ij]** ↵ 6. **[lock]** ↵ -> select sample solvent 7. **[bsmsdisp]** ↵ to adjust **Z** and **Z2** or do **[gradshim]** ↵	Insert the sample in the magnet. Lock the spectrometer. Readjust the Z and Z2 shims until the lock level is optimized. If the instrument has gradients do gradient shimming. Tune and match the probehead for ^{1}H observation, if necessary.

Figure 2: *TopSpin Window*



Step(s) and command(s)	Comment(s)
 8. Type in the command line **[eda]** ↵, **PULPROG = zg**, type **solv** ↵ in the search window and press enter on keyboard; select the appropriate solvent and click on , or type in the command line **[getprosol]** ↵).	You could type each parameter in the command line and set the value. **[eda]** ≡ AcquPars **PULPROG** ≡ Current pulse program The command ≡ **[getprosol]** ↵ , set the some acquisition parameters like pulse values.
9. Type in the command line **[d1]** = 10 ↵, **[rga]** ↵ to adjust the receiver gain, then **[ns]=1** ↵, **[ds]=0** ↵ and **[zg]** ↵ 10. **[ef]**, **[apk]**; phase it if necessary.	**[d1]:** relaxation delay; 1-5*T1 **[rga]**: Adjust receiver gain **[ns]**: to select the number of acquisitions **[ds]**: to dummy scans and **[zg]**: to acquire the 1D proton spectrum
11. Set **o1** on resonance with , then **[swh] = 500** 12. **[TD] =1K ; [lb] =2**↵ ; **[ro]**↵ , then click on **Stop rotation** ; **[ns] = 8** ; **[ds] = 2** ; **[zg]** 13. **[ef]** ; **[apk]** ; confirm that no saturation, artifacts, etc. are observed.	
14. **[iexpno]** ↵ 15. **[eda]** ; **[pulprog] = cpmg** ; Switch from 1D to 2D clicking on ; **[fnmode] = QF** ; **[aqmod] = qsim** or **DQD** (will depend on the instrument under use). 16. **[td]**↵ **F2 = 1024 F1 = 10** 17. **[ns] = 8** ; **[ds] = 2** ; **[d1] = 1-5 * T1** 18. **[d20] = 10m** ; **[d11] = 30m** ; **[L4] = 10**	From the previous data set create the new data set for the T_2 experiment. Also switch from 1D to 2D. **[d1]**: relaxation delay; You should know the T_1 or an approximate value. The value for **L4** will depend on the values entered in the **vc** list

19. **[edlist]** ↵, select **vc** (Variable counter) , select **t2delay** and click **Ok**. Enter the values as shown below: 2 20 50 100 200 300 400 500 750 1000 **Click OK** (means save it) 20. **[zg]**	IMPORTANT: The values shown are only an example; you have to set the values that better work for you.
21. **[edp]** ↵ **[si]**↵ F2 = 512 F1 = 16 **[wdw]**(F2) = EM **[lb]** = 2 **[phmod]** = no **[pknl]** = true **[bcmod]** for F2 = **quad**, for F1 = **no** **[mc2]** = **QF**	**[edp]** ≡ ProcPars You could type each parameter in the command line and set the value.
22. **[xf2]** ↵ 23. Click on  , and select at least two rows. Do a phase correction to that rows and store the correction (). Then 	Save and return:  Return: 
24. Before any calculation open the home directory and in **Location** type the directory where is your T_2 experiment file. For example, **Location:** file: /opt/topspin/data/user/nmr/NAME/EXPNO In that Directory right click the mouse and select **Create New → Text File**, then name it **vdlist**. Open that file and enter the following list 0.04 0.4 1 2 4 6 8 10 15 20	**user**: nmr in our case **NAME:** filemane of the experiment **EXPNO**: experiment number IMPORTANT: The values shown are the delay values derived from the vclist.

25. **Analysis** → **T1/T2 Relaxation [t1guide]** 26. Click on Extract → Spectrum → Slice: 1  27. Click on **Define Ranges** and select the signal ( ) to which the T_2 will be obtained. 28. Click on to save as **Export Regions to Relaxation module and .ret**.	
29. Click on **Relaxation Window**, select the **Fitting Function** → **Function Type** → **expdec**. Confirm that the **vdlist** (List File Name) is selected too and click on Apply then Ok.	**expdec**: I[t]=I[0]*exp(-t/T)
30. Select **Fitting Type** → **Area** and click on **Start Calculation**. 31. Click on **Display Report**, then **Print**.	

References:

1. **TopSpin Users Guide**, Part Number H9469SA1 V2/April 1th 2004
2. 150_and_more_v2.pdf.