University of Puerto Rico
NMR LAB
STANDARD OPERATING PROCEDURE: NMR-01

TITLE: **Proton T_1 Measurement**		
Original Issue: 08 DEC 2005	Revision Date: 5 JUN 2006	**Page 1 of 5**

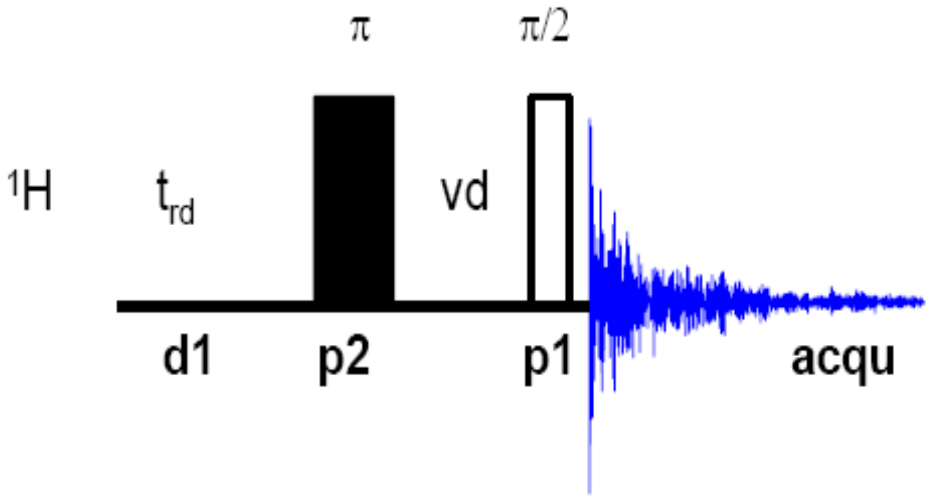
Prepared By: José R. Martínez	Approved By: José R. Martínez

Introduction

The spin-lattice relaxation time of t he various ^1H nuclei of a molecule may be determined by using the inversion recovery pulse sequence. The pulse sequence begins with a recycle delay (t_{rd}) that is sufficiently long to ensure that all magnetization returns to equilibrium (i.e., pure z-magnetization). A 180° pulse is applied which inverts the magnetization. The recovery delay follows to allow varying degrees of T_1 relaxation (depending on the value of the recover y delay time). The final 90° pulse then converts any z-magnetization into observable transverse magnetization, which is detected during the acquisition period immediately following the final pulse.

We assume that the main program (TopSpin) to operate the instrument and the lock display (**[lockdisp]** ↵) were activated previously.

***Figure 1**: Inversion Recovery Pulse Sequence*



Procedure

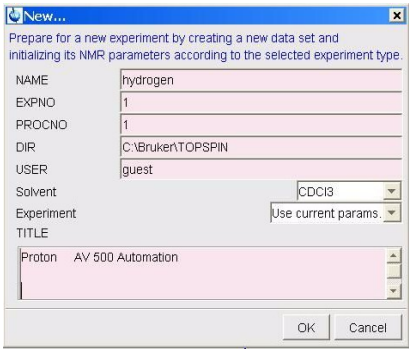
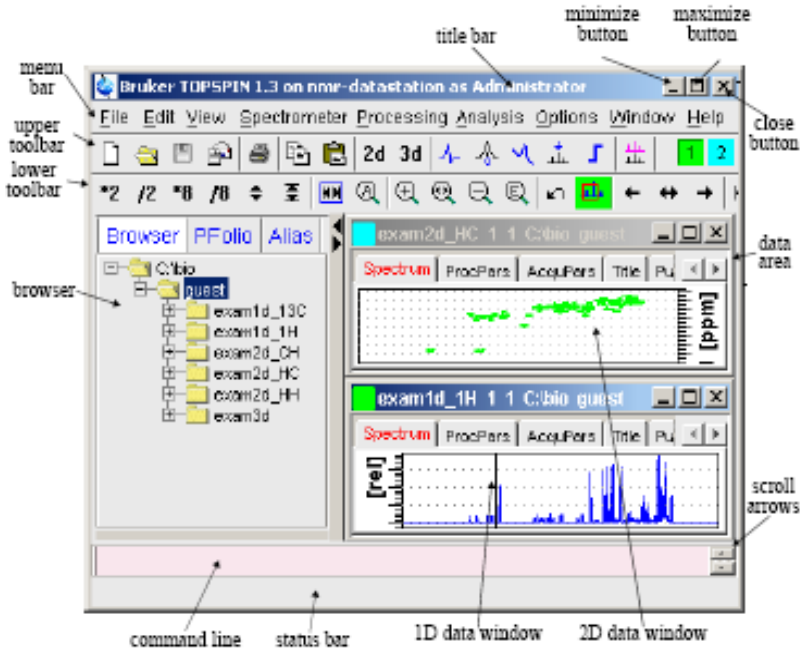
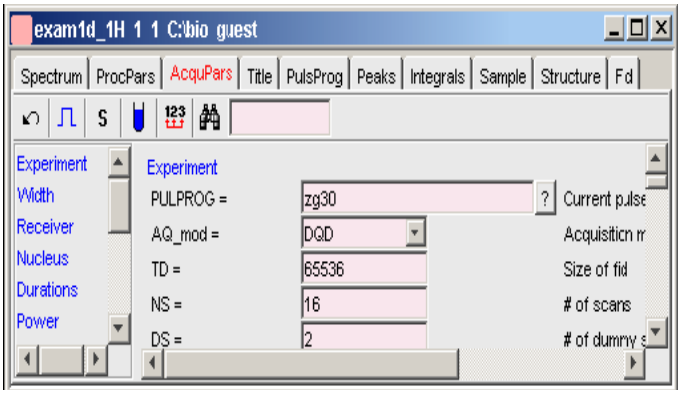
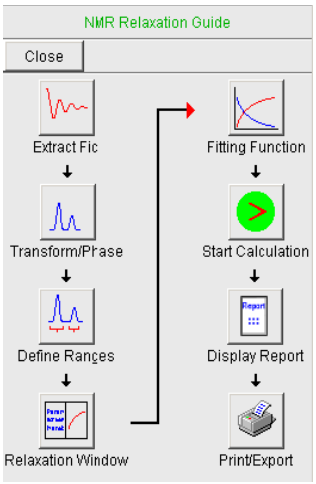
Step(s) and command(s)	Comment(s)
1. **[edc]** ↵  	Create the data set to record a ^{1}H reference spectrum. ↵ : means press enter in the keyboard. Important: DIR= **/opt/topspin** USER= **NMR** Experiment= PROTON
In the command line (see Figure 2) type 2. **[ej]** ↵ (to take out the previous sample) 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 (≡ **[getprosol]** ↵).	**[eda]** ≡ AcquPars **PULPROG ≡ Current pulse program**
9. Type in the command line **[rga]** ↵ to adjust the receiver gain, then **[d1]=5** ↵, **[ns]=8** ↵, **[ds]=2** ↵ and **[zg]** ↵	**[rga]**: Adjust receiver gain **[d1]**: relaxation delay; 1-5*T1 **[ns]**: to select the number of acquisitions **[ds]**: to dummy scans and **[zg]**: to acquire the 1D proton spectrum
10. **[iexpno]** ↵ 11. In the acquisition window (click on **AcqPars** or type **[eda]** in the command line), **PULPROG= t1ir**. Then, click on 123 to covert that file from 1D to 2D.	From the previous data set create the new data set for the T_1 experiment. Also switch from 1D to 2D.
12. **[edlist]** ↵, select **vd** , select **t1delay** and click **Ok**. Enter the delays in seconds as shown below: 10 5 4 3 2 1 0.5 0.25 0.1 0.01 **Click OK** (means save it)	The values shown are only an example; you have to set the values that better work for you.

13. **[eda]** ↵ , In the **command line** type each parameter shown below to set the value. **[pulprog]** = t1ir **[td]** for F2 = 16k, for F1 = 10 **[ns]** = 8 **[ds]** = 4 **[d1]** = 10s **[L4]** = 10 **[vdlist]** = t1delay Then, type **solv** ↵, confirm that the solvent is correct and click on  to confirm that the pulse values are correct too. 14. **[zg]**	Type **[eda]** if necessary; maybe you should be already in that window. The command  ≡ **[getprosol]** ↵ , set the some acquisition parameters like pulse values; remember that the value for **L4** will depend on the values entered in the **vd** list. **VDLIST**= t1delay list or the filemane for the list that you created. To select the list click on the **?** icon.
15. **[edp]** ↵ **[si]** for F2 = 8k, **[si]** for F1 = 16 **[wdw]** = 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.
16. **[xf2]** ↵ 17. Click on  , and select two rows. Do a phase correction to that rows and store the correction (). Then 	Save and return:  Return: 

18. **Analysis** → **T1/T2 Relaxation [t1guide]** 19. Click on Extract → Spectrum → Slice: 1  20. Click on **Define Ranges** and select the signal(s) ( ) to which the T1 will be obtained. 21. Click on to save as **Export Regions to Relaxation module and .ret.**	
22. Click on **Relaxation Window**, select the **Fitting Function** → **invrec**. Confirm that **vdlist** is selected too and click on Ok.	**invrec**: I[t]=I[0](1-2*A*exp(-t/T1))
23. Select **Fitting Type** → **Area** and click on **Start Calculation**.	
24. Click on **Display Report**, then **Print**.	

References:

1. **TopSpin Users Guide**, Part Number H9469SA1 V2/April 1th 2004
2. **Avance 1D and 2D Course**, April 1, 2003, Bruker AG Fällanden, Switzerland, Version 030401 Chapter 19, page 148.