Acquiring solid state NMR data on the Bruker 500 Avance system at UVM

By Monika Ivancic, October, 2014, edited Oct. 22, 2015

1. Pack your sample into the rotor properly (see Bruker instruction video: http://www.theresonance.com/nmr-tutorial-mas-rotor-filling/). Double check the cap to make sure it is on tight (if you need to remove/change the cap use the 'cap removal tool'). Check to make sure that the Sharpie mark is thick enough.

2. To insert packed rotor: remove the 'sample trap' at the top of the transfer tube. Drop your sample into the transfer tube, with the cap pointing up (ie. Bottom down) and replace the 'sample trap' ensuring that it is closed.

3. In the MAS Pneumatic Unit Control (ie. MAS GUI) within TopSpin, click 'insert' and wait until it turns from green to grey; enter the desired MAS rate (depends on which nucleus acquiring, generally 15kHz for ^{13}C, 8kHz for ^{31}P or ^{29}Si) and click 'go' (takes about 60s or so to get to desired rate)
   If MAS won't spin up: click 'halt' then remove sample (click 'eject' & sample will shoot up into 'sample trap') and check the rotor cap, also check the Sharpie mark; repeat no. 3

4. In Topspin, create a new data set: type 'new' or go to Start -> Create Dataset. Enter sample name, expno (start with 1) and procno (start with 1). Browse for the desired experiment (all the solids parameter sets will start with 'uvmss'), don't define solvent nor click on getprosol, nor on 'keep current parameters'. Make sure your data will be stored under /home/username(yours)/data. Then click OK. Your new data set is now created.

5. You will need to manually tune the probe to the desired frequency (^{13}C, ^{29}Si, ^{31}P, …) and check ^{1}H tuning. In the data set that you just created, type 'wobb', which displays the 'tune and match' curve. Either turn the screen so that you can see it from the bottom of the magnet OR use the display ontop of the preamp. The blue T (tune) and M (match) screws are for adjusting the X-nucleus. Start with centering the dip with T, then make the dip touch the bottom with M. These are interdependent, so you will have to go back and forth to optimize the dip at the center. Once you're done with the X-nucleus, click 'next' either in TopSpin OR on the display above the preamp. This should switch to the ^{1}H nucleus tuning. The yellow T and M are for ^{1}H tuning. Follow the same procedure as with the X-nucleus. Use the red screwdriver tool to adjust these screws. Once you've optimized both nuclei, click on the Stop icon in TopSpin.

6. To do any solid state experiment, you will first need to know the T1 relaxation time of the ^{1}Hs in your sample. You will need to know this, so that you can set the D1 relaxation delay (time for spins to relax) in your experiment (cp, cppi, cpnqs, cptoss, etc.). So start with the

"uvmss.1HT1measure" parameter set and acquire the data on your sample. Use the 'xf2' command to process this data.

7. Analyzing the T1 data: begin with ensuring the spectrum is phased properly (peaks are all positive absorption peaks). 'apk' is quite good, but for manual touch up go to Process -> Phase correction (see Monika if you need help with this).
   Analyze -> T1/T2 module (and follow flow of buttons here):
   Fid: in GUI choose either Fid or Spectrum and enter a slice number
   (8 thru 16 have more signal, so choose one of these)
   Peaks/Ranges: in GUI choose 'manual peak picking' (and enter 'peak picking' mode)
   Choose the largest 3 to 5 peaks (depending on your sample)
   Click the 'save A' icon and choose the 3rd option in the drop-down
   Relaxation: click icon and in GUI select fitting function (uxnmrt1 for this data)
   Make sure 'list file name' matches data (vdlist), then click 'OK' (exits GUI)
   Click 1st (double arrow) icon, which calculates fit for all selected peaks
   These are the T1 values for your selected peaks; Intensity of each peak vs. tau is plotted and T1 is determined according to: $I(t) = I(0) + P^* \exp(-t/T1)$
   For your solid state experiments you will choose a D1 value ≥ 2* T1 (close the T1 analysis window.)

8. Create a new data set: type 'new' (and increase the experiment number for the same sample) or type 'iexpno' in the command line. Choose the appropriate parameter set from the 'uvmss' list. If you used the 'iexpno' command, you will need to type 'rpar' (read parameters) and find the parameters you need:

9. Parameter sets:
   uvmss.1HT1measure (pseudo-2D for measuring ^{1}H T1 relaxation)
   uvmss.13Ccp (^{13}C-^{1}H cross-polarization expt)
   uvmss.13Ccppi (^{13}C cp, optimized for CH_2 peaks to appear, 4 C's gone)
   uvmss.31Pcp (^{31}P-^{1}H cross-polarization expt)
   uvmss.29Si-cp (^{29}Si-^{1}H cross polarization expt)
   uvmss.1Honepulse (basic ^{1}H experiment)
   uvmss.13Ccpnqs (^{13}C-^{1}H cp with 'non-quaternary suppression')

10. Adjusting parameters: D1 is your main relaxation delay, set to 2* longest T1
    NS number of scans, set to a large number, if you don't know when the signal in your sample will show up
    SW if you would like to change your sweep width (total width)
    O1P if you need a different center of spectrum
    SW and O1P go hand in hand and together will determine range
    AQ may not be above 50 msec, so if reducing the SW makes this value go above 50 msec, you will need to reset it to 50 msec (or 0.05 sec)

11. Acquiring your data: RG or 'receiver gain' may be adjusted prior to acquiring with 'rga' (for many of the X-nculei it is set to 128, which usually works) ZG or clicking the green arrow, will proceed to acquiring your data

12. Processing your data: you may use 'efp' followed by 'apk' to process See Monika if you need further processing help or use another program such as MestreNova or SpinWorks

13. Ejecting your sample: on the MAS GUI, you will stop the sample spinning by clicking 'halt'. Wait until spinning is down to 0 before clicking 'eject'. The sample should pop out into the sample trap. Take the sample trap off the top of the Sample Transport Tube to retrieve your sample.