

## Setting Up CP/MAS experiments for “X” channel nuclei (eg. $^{13}\text{C}$ , $^{27}\text{Al}$ , $^{15}\text{N}$ )

The “standard” samples for setting up CP/MAS experiments are as follows:

**For  $^{13}\text{C}$**  = adamantane or hexamethylbenzene (HMB). Adamantane is the preferred sample for  $^{13}\text{C}$  CP since it has a very narrow Hartmann-Hahn match. However, when setting up CP/MAS using the 4mm probe with very fast spinning speeds (spinning speed > 10 kHz), it is very difficult to find the H-H match so HMB is used instead. An alternative sample for the 4mm probe can be L-tyrosine hydrochloride

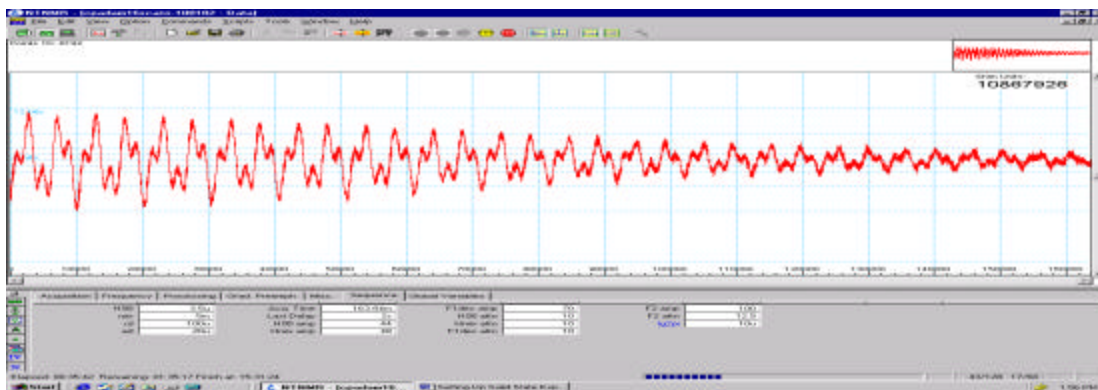
**For  $^{29}\text{Si}$**  = Kaolin

**For  $^{15}\text{N}$**  =  $^{15}\text{N}$  labeled glycine or  $^{15}\text{N}$  labeled alanine

**For  $^{27}\text{Al}$**  = aluminum oxide ( $\text{Al}_2\text{O}_3$ )

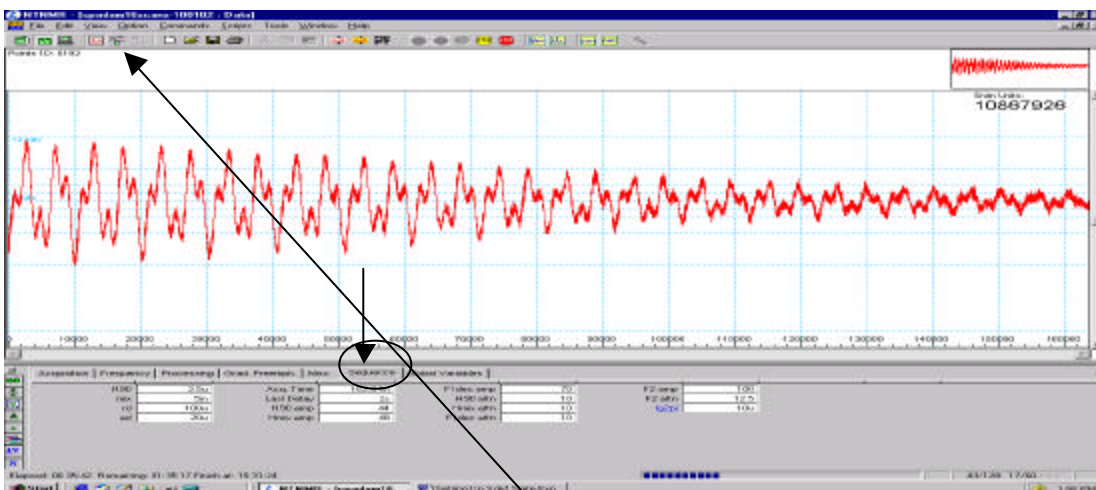
### To set up $^{13}\text{C}$ CP/MAS:

- 1) Put sample in probe and start it spinning. It is best to spin the standard sample at the same speed you will run your experiment. For the Bruker probe, use < 6 kHz; for the 7mm Chem Magnetics probe use 4 kHz; and for the 4 mm Chem Magnetics use 17 kHz
- 2) Open up the  $^{13}\text{C}$  calibration file from the appropriate directory in the E: E:/data/calibrations/13C/appropriate probe(4mm chem,bruker, etc.)/cpadam16scans-most current date
- 3) Tune the probe as outlined in MAS NMR section (remember  $^{13}\text{C}$  observe is channel 2)
- 4) Take 16 scans with the previous settings and check the signal intensity. The FID should look similar to the following

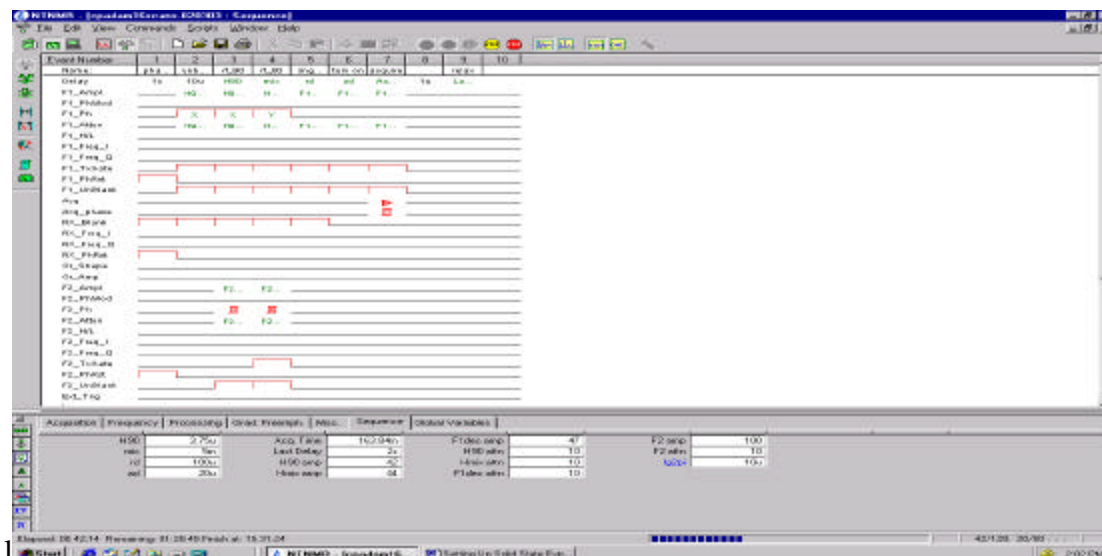


The FID should ring out and the rotational echoes should be observable throughout the window.

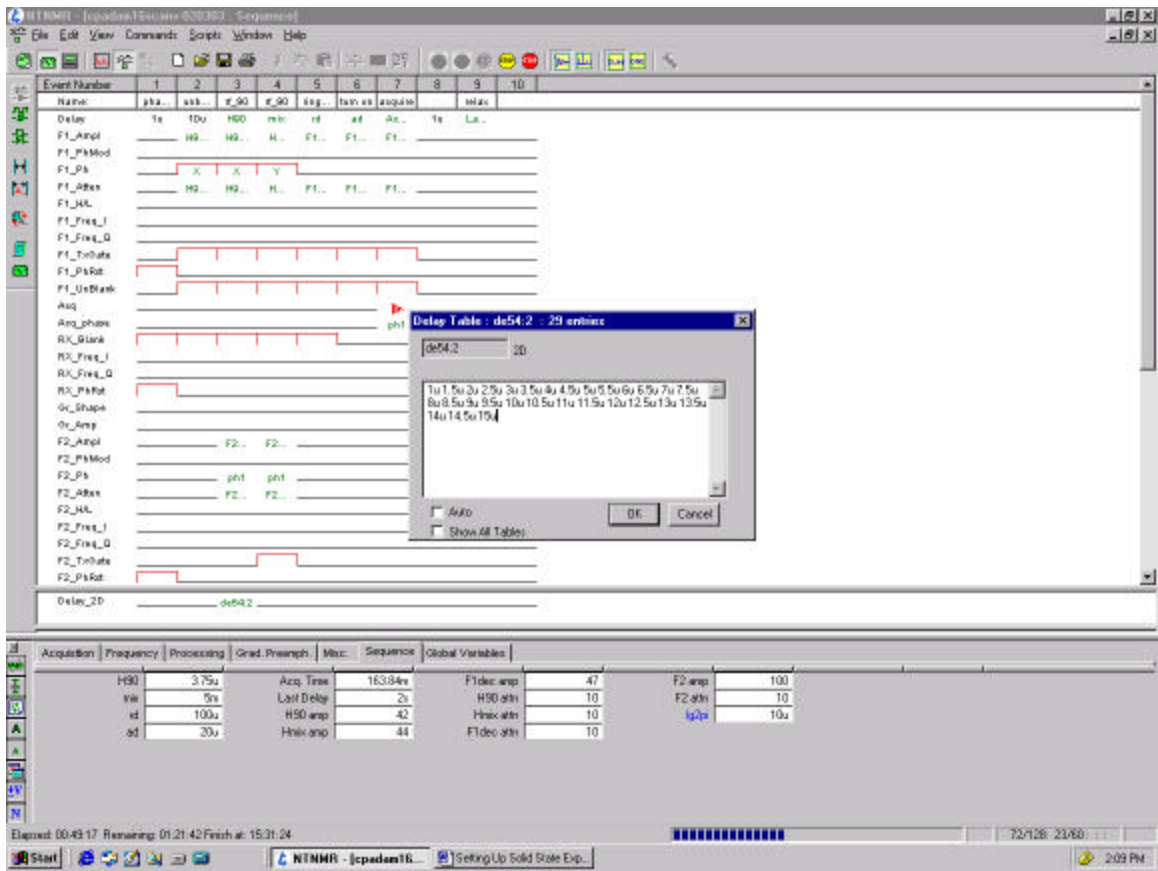
- Next, you want to check the 90° pulse on the  $^1\text{H}$  channel. To do this click on **“Sequence”** bar under the spectrum



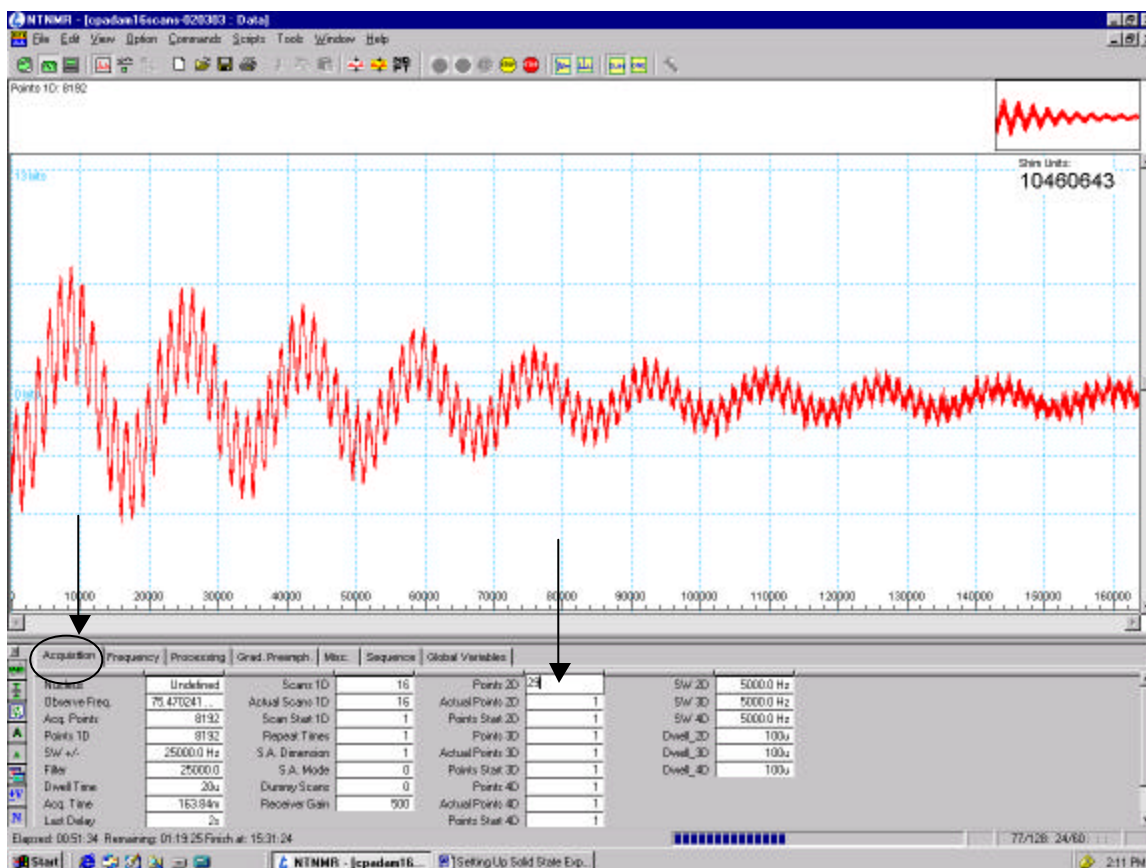
next click on the pulse sequence Icon located on the top left corner



click on **“H90”**, then select **“2D table”**, **right click** on the icon that appears and enter values in the table. Typical arrays are between 1-15us in steps of .5 us.



click “ok” to set the array. Then click on “Acquisition” and enter in the correct value under “Points 2D”



- 6) Once the PW array is completed, note the 180° and 90° pulse. An appropriate 90° pulse is between 3-6 us. Click on the “**Sequence**” icon to place the correct value for pw in the box “**H90**”. The power levels for H90 are controlled by “**H90 amp**” and “**H90 attn**”.
- 7) Next step is to optimize the <sup>1</sup>H power for the Hartmann-Hahn match. This parameter is controlled by “**Hmix amp**” and “**Hmix attn**”. Change the value of “**Hmix amp**” and note the difference in signal intensity. Look for the value that gives you the best signal intensity after 16 scans. Since the H-H match for adamantane is very narrow the difference of 1 (i.e. 43- 44 for Hmix amp) can have a big influence on signal intensity so change the values by increments of 1 or 0.5.
- 8) After the H-H power is optimized, the decoupling power can be adjusted in similar manner. The power for the decoupling strength is controlled by “**F1dec amp**” and “**F1dec attn**”.
- 9) After the optimization is performed, make sure all the appropriate power levels and delays are set correctly, change the number of scans to the desired number and set the **receiver gain**. For samples that consist of materials supported on a surface set the receiver gain to 2000. For pure samples such as organics or

starting materials set the gain between 100-1000. Create a new file by selecting “**Save As**” under the “**File**” window. Make sure the target directory is correct before saving the file.