^{13}C CP/MAS NMR

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Brief Summary

This document is specific at this time to performing "normal" (5-8 kHz) and "fast" (9-18 kHz) magic-angle spinning experiments using the 4mm Doty probe [i.e., it does not yet have information specific to the 1.2mm Phoenix probe, which can spin "very fast" (up to 60 kHz)]. Spinning sidebands (SSBs) will overlap in the standard ^{13}C chemical shift range for normal spinning. CP/TOSS, a technique to reduce the SSBs, will often be the preferred experiment when spinning < 12 kHz [I have yet to investigate the utility of SELTICS as an alternative to TOSS.] When spinning fast, standard CP/MAS should be sufficient to gain chemical shift information.

Note that to spin fast, you must use a thickwall rotor. Thinwall rotors can spin up to ≤ 12 kHz. You must also be using proper caps. Currently I recommend the glass-filled torlon (GFT) caps for fast spinning (***not*** the pure torlon rotors). Kel-F caps can be spun up to only 9kHz.

A. ***Initial Setup:***

1. Pack the rotor and check that it spins up to at least 5 kHz ok. The cap tip always goes down; drive cap goes up.

   When using the bench spinner, ***always*** keep the bearing air high enough, and match it with the drive air pressure according to the sheet next to the bench spinner. Failure to maintain the bearing air at proper pressures can damage the probe!

2. If you use the prep area in the facility, leave it clean!

3. It is a good idea to check the magic-angle in the probe using the KBr in the KBr+adamantane sample. This procedure does not need to be done every time, but all ssNMR users should know how to do this.

4. Adamantane provides a good chemical shift reference for ^{13}C. Since all experiments are run unlocked, some type of reference is needed. Unless you know better, take a quick ^{13}C adamantane spectrum and reference the upfield peak to 28.7 ppm.

   For KBr and adamantane checks, log your values in the spreadsheet kept at:

   /home/shared_docs/SSNMR_MAS_log.xls

   If the SR value for adamantane is set prior to the data being obtained, it will be stored in the castor archived data and will come in automatically into MNova. If it is stored after the data is obtain, copy the data from the spectrometer back to castor.

5. Put in your sample and get it to spin up to the proper speed. Check that the rotor and caps are compatible with the speed you are targeting (i.e., do not spin too fast!).

6. Use the following parameter sets:

   CP/MAS: 4mm_C13_cpmas.gUW

   CP/TOSS: 4mm_C13_cptoss.gUW (only for mas ≤ 8kHz)

7. Update the parameters to correct for the chosen spinning speed using the python script:

   cp_4.0mm↵ ;you can also enter **edpy** select that script and click EXECUTE

You must enter the correct spinning speed.

Enter all the values as shown, unless you know to do otherwise.

The other parameter most often be changed here is **P15**, the contact time. It is common to run multiple experiments with varying **P15** to verify the maximum cross-polarization. Aromatics typically optimize to longer contact times than aliphatic carbons, as an example. The optimum value depends on the cross-relaxation of the carbons, and on the $T_{1\rho}$ of the protons. Measurement of $T_{1\rho}(^1H)$ is also regularly done (look for future documentation on this) when more complex experiments need to be done.

8. **wobbt↵** ;tune to ^{13}C, and to ^1H. Make sure that the preamp box shows no yellow lights.
   ;switch between channels by pressing F2 on the preamp 2 or 3 times
9. Check that the lock is off: **locnuc↵**
10. Set SR properly from adamantane reference. If SR is set after acquisition, copy the data to castor.
11. **zg↵** ;use **tr↵** to check s/n, and halt on multiple of 8 scans.
11. Don't apply 1st order phase corrections. **apk0↵** applies only 0 order; don't use **apk** or **efb**.