

13C CP/MAS solid-state NMR at SSNMR 500 MHz

Procedure:

Open Topspin 4.0.7

1. pack sample into **3.2 mm** rotor: <https://www.youtube.com/watch?v=bNFJj2g0UjI>
2. under "**group name**" folder in Topspin, drag demo/CP dataset
3. create dataset: name; exp #; pro #; directory (D:**group name**\user last name\yrmo); no solvent
4. open **MAS control unit**
5. insert sample into magnet: the solid-state NMR sample loading line is different from solution NMR; click (**insert**) & wait until the sample is inside (~10 s)
6. start (click **go**) magic-spinning to **4 kHz (Set in MAS unit)** until the spinning rate is stable (**green number**)
7. start tuning/matching (type **wobb**); go to adjust 13C tuning/matching knobs under the magnet; switch to 1H nuclei in the tuning/matching window and adjust 1H tuning/matching knobs under the magnet. The step needs to repeat for the 1st nuclei if the 2nd nuclei tuning/matching position has changed.
8. spinning up to the target spinning rate by changing **Set** in MAS unit: increase **1kHz** step by step until the spinning rate is stable (**green number**)
9. check: (**ns**) (**1k**), (**d1**), (**ds**)
10. (**zg**)
11. check spectrum sensitive: (**tr;ef;pk**); if the spectrum sensitive is enough, type (**halt;ef;pk**)
12. or wait until experiments finish, type (**ef;pk**); if the spectrum sensitive is not enough, change (**ns**) to the desired number, type (**go**)
13. adjust phase if necessary
14. calibrate spectrum
15. pick peaks
16. change spinning to 4 kHz by reducing **1kHz** step by step; stop spinning
17. eject sample (**ej**); take sample
18. close topspin
19. log off