13-C NMR Protocol for beginners
AV-400

1. Follow procedure for collecting 1H NMR through the print step, then collect 13C NMR
2. Record run in logbook

Preparation for Data Aquisition

3. Drag 13-C folder from your research group subdirectory (in the "NMR Data Browser" screen) to the "Bruker TOPSPIN" window
4. Go to the "file" tab in the "Bruker TOPSPIN" window and choose "new"
5. Name the file (do NOT use long name or any special characters), choose the exp # and solvent type. Click "ok"

Data Aquisition and Processing

6. Initialize instrument by typing **ii** into "Bruker TOPSPIN" window and hit enter
7. Tune and match the probe by typing **atma** into "Bruker TOPSPIN" window and hit enter (wait until finished to proceed)
8. Make sure SPIN is still on
9. Adjust the receiver gain by typing **rga** into "Bruker TOPSPIN" window and hit enter
10. Type **ns** into "Bruker TOPSPIN" window (consult with your group members for how many scans to start with)
11. Type **ds** into "Bruker TOPSPIN" window and choose 4 as the setting
12. Type **d1** into "Bruker TOPSPIN" window and choose delay time (start with 0.7 sec and increase up to 5 sec as needed)
13. To acquire data type **zg** into "Bruker TOPSPIN" window and hit enter
14. To process data type **efp;apk** into "Bruker TOPSPIN" window, hit enter, and wait for spectrum to appear
15. You can check the spectrum before the run is over by typing **tr;efp;apk** into "Bruker TOPSPIN" window. You can stop the run if desired by typing **halt** into the "Bruker TOPSPIN" window. You can add more scans if needed by adjusting **ns** and typing **go** to start
16. Click on the "spectrum calibration" icon in the "Bruker TOPSPIN" window and choose the solvent peak (e.g. set chloroform to 77.0)
17. Click on the "manual peak picking" icon in the "Bruker TOPSPIN" window and label the desired peaks and then click on the "return, save changes" icon
18. Print the data

Final Steps

19. Turn off the SPIN, turn off the LOCK (both on BSMS panel window). Wait until “finished”
20. Turn on sample LIFT on BSMS panel window
21. Change the sample in the spinner to the standard D2O sample (use the depth gauge), wipe the tube and spinner with a kimwipe and insert into NMR
22. Turn off sample LIFT on BSMS panel window
23. Replace cap on NMR
24. Click on the "lock display" icon in the "Bruker TOPSPIN" window
26. Click on the "lock current sample" icon in the "lock display" window
27. Double click on D2O for the standard sample solvent
28. Enter end time in logbook