

13-C NMR Protocol for beginners DRX-400

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

Preparation for Data Acquisition

3. Drag 13-C folder from your research group subdirectory (in the "NMR Data Browser" screen) to the "Bruker TOPSPIN 1.3" window
4. Go to the "file" tab in the "Bruker TOPSPIN 1.3" 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"
6. Turn off sample SPIN on BSMS panel
7. Type **wobb** into "Bruker TOPSPIN 1.3" window
8. Adjust matching (up & down) and tuning (left & right) as needed (use slide numbers-LAST digits only)
9. Click on the "switch to next channel/nucleus" icon and adjust matching and tuning as needed (only 13C if already did 1H earlier)
10. Click on STOP icon when finished
11. Turn on SPIN on BSMS panel

Data Acquisition and Processing

12. Type **ns** into "Bruker TOPSPIN 1.3" window (consult with your group members for how many scans to start with)
13. Type **ds** into "Bruker TOPSPIN 1.3" window and choose 0 as the setting
14. Type **d1** into "Bruker TOPSPIN 1.3" window and choose delay time (start with 0.695 sec and increase up to 5 sec as needed)
15. Type **ii;rga;zg;efp;apk** into "Bruker TOPSPIN 1.3" window and wait for spectrum to appear
16. You can check the spectrum before the run is over by typing **tr;efp;apk** into "Bruker TOPSPIN 1.3" window. You can stop the run if desired by typing **halt** into the "Bruker TOPSPIN 1.3" window. You can add more scans if needed by adjusting **ns** and typing **go** to start
17. Click on the "spectrum calibration" icon in the "Bruker TOPSPIN 1.3" window and choose the solvent peak (e.g. set chloroform to 77.0)
18. Click on the "manual peak picking" icon in the "Bruker TOPSPIN 1.3" window and label the desired peaks and then click on the "return, save changes" icon
19. Print the data

Final Steps

20. Turn off SPIN, Turn off LOCK (Both on BSMS panel)
21. Turn on sample LIFT on BSMS panel
22. Change the sample in the spinner to the standard sample (use the depth gauge), wipe tube and spinner with kimwipe and insert into NMR
23. Turn off sample LIFT on BSMS panel
24. Replace cap on NMR
25. Click on the "lock display" icon in the "Bruker TOPSPIN 1.3" window
26. Click on the "lock current sample" icon in the "lock display" window
27. Double click on D2O for the standard sample
28. Enter end time in logbook