

13C NMR Protocol for Beginners DPX-300/Avance-300

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

Preparation for Data Collection

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".

Data Acquisition and Processing

6. Type **ns** into "Bruker TOPSPIN 1.3" window (consult with your group members for how many scans to start with)
7. Type **ds** into "Bruker TOPSPIN 1.3" window and choose 4 as the setting
8. 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)
9. Type **ii;rga;zg;efp;apk** into "Bruker TOPSPIN 1.3" window and wait for spectrum to appear
10. 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
11. Click on the "spectrum calibration" icon in the "Bruker TOPSPIN 1.3" window and choose the solvent peak (e.g. set chloroform peak to 77.0)
12. 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
13. Print the data

Final Steps

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