

1H NMR Protocol for Beginners DPX-300/Avance-300

1. Enter run in logbook

Sample Change

2. Open TopSpin 1.3 program
3. Drag 1-H folder from your research group subdirectory (in the "NMR Data Browser" screen) to the "Bruker TOPSPIN 1.3" window
4. Remove cap from NMR
5. Turn off LOCK on BSMS panel
6. Turn off sample SPIN on BSMS panel if it is on (it should be off already if the standard sample is in the instrument)
7. Turn on sample LIFT on BSMS panel
8. Take standard sample out of NMR and replace your sample into spinner (make sure to use the depth gauge)
9. Wipe the NMR tube and spinner with a kimwipe
10. Insert sample into NMR
11. Turn off sample LIFT on BSMS panel

Preparation for Data Acquisition

12. Go to the "file" tab in the "Bruker TOPSPIN 1.3" window and choose "new"
13. Name the file (do NOT use long name or any special characters), choose the exp # and solvent type. Click "ok"
14. Type **rsh std.shim** in the "Bruker TOPSPIN 1.3" window
15. Click on the "lock display" icon in the "Bruker TOPSPIN 1.3" window
16. Click on the "lock current sample" icon in the "lock display" window
17. Double click on the solvent that you are using and wait for the message box in the "Bruker TOPSPIN 1.3" window to say "lock finished"
18. Turn on SPIN on BSMS panel
19. Turn on Z1 on BSMS panel and turn wheel to adjust signal to highest level
20. Turn on Z2 on BSMS panel and turn wheel to adjust signal to highest level
21. Repeat steps 19 and 20 until signal is at highest level possible (always end with adjusting Z1). If signal goes off screen, press the LOCK GAIN button on the BSMS panel and turn the wheel until you can see signal again then turn on Z1 again and continue shimming.
22. Press STDBY button on BSMS panel

Data Acquisition and Processing

23. Type **ns** into "Bruker TOPSPIN 1.3" window (start with 16 scans)
24. Type **ds** into "Bruker TOPSPIN 1.3" window and choose 0 as the setting
25. Type **d1** into "Bruker TOPSPIN 1.3" window and choose delay time (start with 0.01)
26. Type **ii** into "Bruker TOPSPIN 1.3" window
27. Type **rga;zg;efp;apk** into "Bruker TOPSPIN 1.3" window and wait for spectrum to appear
28. Click on the "spectrum calibration" icon in the "Bruker TOPSPIN 1.3" window and choose the solvent peak (e.g. set chloroform to 7.26)
29. 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
30. Click on the "interactive integration" icon in the "Bruker TOPSPIN 1.3" window, then click on the "define integral regions" icon on the submenu and choose the desired integration areas. Click on the "return, save changes" icon when finished
31. Print the data

Final Steps

32. Turn off SPIN, Turn off LOCK (both on BSMS panel)
33. Turn on sample LIFT on BSMS panel
34. Change the sample in the spinner to the standard sample (use the depth gauge) wipe tube and spinner with kimwipe, insert into NMR
35. Turn off sample LIFT on BSMS panel
36. Replace cap on NMR
37. Repeat steps 15 and 16 and double click on D2O for the standard sample
38. Enter the end time on the logbook