1H NMR Protocol for Beginners
DRX-400

1. Record 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 not be on if standard sample is in NMR)
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), wipe the tube and spinner with a kimwipe
9. Insert sample into NMR
10. Turn off sample LIFT on BSMS panel

Preparation for Data Aquisition
11. Go to the "file" tab in the "Bruker TOPSPIN 1.3" window and choose "new"
12. Name the file (do NOT use long name or any special characters), choose the exp # and solvent type. Click "ok"
13. Type **rsh std.shim** in the "Bruker TOPSPIN 1.3" window
14. Click on the "lock display" icon in the "Bruker TOPSPIN 1.3" window
15. Click on the "lock current sample" icon in the "lock display" window
16. 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"
17. Turn on SPIN on BSMS panel
18. Turn on Z1 on BSMS panel and turn wheel to adjust signal to highest level
19. Turn on Z2 on BSMS panel and turn wheel to adjust signal to highest level
20. Repeat steps 18 and 19 until signal is at highest level possible (always end with adjusting Z1). If the signal goes off the screen, press the LOCK GAIN button on the BSMS panel and turn the wheel until you can see the signal again, then turn on Z1 again and continue shimming.
21. Press STDBY button on BSMS panel
22. Turn off sample SPIN on BSMS panel
23. Type **wobb** into "Bruker TOPSPIN 1.3" window
24. Make sure the correct nucleus is selected (^{1}H). Click on the "switch to next channel/nucleus" icon if needed. Adjust matching (up & down) and tuning (left & right) as needed (silver pegs) and then click on STOP icon when finished
25. Turn on SPIN on BSMS panel

Data Aquisition and Processing
26. Type **ns** into "Bruker TOPSPIN 1.3" window (start with 16 scans)
27. Type **ds** into "Bruker TOPSPIN 1.3" window and choose 0 as the setting
28. Type **d1** into "Bruker TOPSPIN 1.3" window and choose delay time (start with 0.01)
29. Type **ii** into "Bruker TOPSPIN 1.3" window
30. Type **rga;zg;efp;apk** into "Bruker TOPSPIN 1.3" window and wait for spectrum to appear
31. 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)
32. 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
33. Click on the "interactive integration" icon in the "Bruker TOPSPIN 1.3" window and choose the desired integration areas and then click on the "return, save changes" icon
34. Print the data

Final Steps

35. Turn off SPIN, Turn off LOCK (both on BSMS panel)
36. Turn on sample LIFT on BSMS panel
37. Change the sample in the spinner to the standard sample (use the depth gauge), wipe the tube and spinner with a kimwipe and insert into NMR
38. Turn off sample LIFT on BSMS panel
39. Replace cap on NMR
40. Repeat steps 14 and 15 and double click on D2O for the standard sample
41. Enter end time in logbook