1H NMR Protocol for Beginners
AV-400

1. Record run in logbook.

Sample Change
2. Open TopSpin program
3. Drag 1-H folder from your research group subdirectory (in the "NMR Data Browser" screen) to the "Bruker TOPSPIN" window
4. Click on “BSMS Panel” icon (opens new window)
5. Turn off LOCK on “main” tab of BSMS panel window (Red means OFF; Green means ON; Grey is ambiguous-CHECK)
6. **MAKE SURE SPIN IS OFF!** Turn off sample SPIN on BSMS panel window if it is on (it should not be on if a standard sample is in NMR) (If Grey-CHECK)
7. Remove cap from NMR
8. Turn on sample LIFT on BSMS panel window
9. Take standard sample out of NMR and replace your sample into spinner (make sure to use the depth measuring device), wipe the tube and spinner with a kimwipe
10. Insert sample into NMR
11. Turn off sample LIFT on BSMS panel window

Preparation for Data Acquisition
12. Go to the "file" tab in the "Bruker TOPSPIN" 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. Lock the field in the “Bruker TOPSPIN” window by clicking on the “lock display” icon and then the “lock current sample” icon and choose correct solvent
15. Initialize instrument by typing **ii** into "Bruker TOPSPIN" window and hit enter
16. Check the temperature by typing **edte** into "Bruker TOPSPIN" window and hit enter (sample temp should equal set temp which should be 298K; if not, make sure probe heat is ON)
17. Tune and match the probe by typing **atma** into "Bruker TOPSPIN" window and hit enter (wait until finished to proceed)
18. Turn on the SPIN on BSMS panel window (wait until button becomes green to proceed)
19. Shim the probe by typing **topshim** into "Bruker TOPSPIN" window and hit enter (wait until finished to proceed)
20. Adjust the receiver gain by typing **rga** into "Bruker TOPSPIN" window and hit enter
21. Enter suitable number of scans, dummy scans, and delay time
    Type **ns** into "Bruker TOPSPIN" window (start with 16 scans)
    Type **ds** into "Bruker TOPSPIN" window and choose 0 as the setting
    Type **d1** into "Bruker TOPSPIN" window and choose delay time (start with 0.01)

Data Acquisition and Processing
22. To acquire data type **zg** into "Bruker TOPSPIN" window and hit enter
23. To process data type **efp;apk** into "Bruker TOPSPIN" window, hit enter, and wait for spectrum to appear
24. Click on the "spectrum calibration" icon in the "Bruker TOPSPIN" window and choose the solvent peak (e.g. set chloroform to 7.26)
25. 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
26. Click on the "interactive integration" icon in the "Bruker TOPSPIN" window and choose the desired integration areas and then click on the "return, save changes" icon
27. Print the data

Final Steps

28. Turn off the SPIN, turn off the LOCK (both on BSMS panel window). Wait until “finished”
29. Turn on sample LIFT on BSMS panel window
30. 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
31. Turn off sample LIFT on BSMS panel window
32. Replace cap on NMR
33. Repeat Step14 and double click on D2O for the standard sample solvent
34. Enter end time in logbook