

¹H NMR Training Protocol for Fourier 300

1. **Record run in logbook** (Date, start time, operator name, funding account, supervisor's name, nuclei, number of samples)
2. Drag 1H folder from your research group's subdirectory (in the "**NMR Data Browser**" screen) to the "Bruker TopSpin 3.1" window (Choose the 1% Ethyl Benzene/CDCl₃ option)
3. Click on the '**Start**' tab in the TopSpin Menu bar and then click on the '**Create Dataset**' icon. Fill out the NAME and EXPNO sections, choose the correct solvent, and then make sure it says PROTON next to the Experiment section. Click OK once you have finished
4. Click on the '**Acquire**' tab and then click the arrow on the '**Spin**' icon. Choose "**turn sample rotation off**"

-----**Sample Related Steps**-----

5. Remove the **magnet cap** from the top of the NMR
6. Click on the arrow on the '**Sample**' icon and choose "**Turn on sample lift air**"
7. Remove the standard sample from the NMR and pull the tube out of the spinner
8. Check the quality of your NMR tube in the NMR tube tester
9. Place your sample in the spinner and then use the depth gauge to push the sample into the spinner to the correct depth. Wipe the spinner and your sample with a Kimwipe
10. Place your sample with the spinner into the NMR
11. Click on the arrow on the '**Sample**' icon and choose "**Turn off sample lift air**"
12. Wait until the sample is lowered into the instrument and the lift air is no longer audible.

13. Click on the '**Lock**' icon, double click on the appropriate solvent and wait until the message box in the lower left hand corner reads "**lockn: finished.**"
14. Click on the arrow on the '**Spin**' icon and choose "**Turn sample rotation on**"
15. Click the '**Shim**' icon (not on the arrow on the 'Shim' icon) and wait until the message box reads "**gradshimau: finished**"
16. Click the '**Prosol**' icon and wait for the message box to read "**getprosol finished!**"
17. Click the '**Gain**' icon and wait for the message box to read "**Job succeeded**"

18. Type "ns" into the lower left hand corner above the message box and press enter. Type the appropriate number of scans and press enter.
19. Click the 'Go' icon. Scanning will now begin. Wait until the message box reads "Job succeeded"
20. Click on the 'Process' tab in the TopSpin Menu bar and click on the 'Proc. Spectrum' icon
21. Zoom in on the peak you would like to set for calibration. Click on the 'Calib. Axis' icon. Click on the center of the appropriate peak, enter the appropriate ppm and hit enter
22. If the phasing is off, click on the 'Adjust Phase' icon and adjust accordingly. Click the 'return, save changes' icon when finished
23. Click on the 'Integrate' icon and highlight individual peaks or the entire spectrum as desired. Click the 'return, save changes' icon when finished
24. Click on the 'Pick Peaks' icon and draw boxes around the desired peaks (top of peak needs to be included in the box). Click the 'return, save changes' icon when finished
25. Click on the 'Publish' tab. You may print the current window by clicking on the 'Print' icon (Dell 2350dn Laser Printer XL should be chosen), or pull up the plot editor by clicking on the 'Plot Layout' icon
26. If you choose to use the plot editor, click the 'open plot editor' icon (this looks like a document with a starburst on it). Make changes as desired and then print (Dell 2350dn Laser Printer XL). Click the 'X' icon to close the Plot editor.
27. You may now run a ^{13}C NMR if desired (refer to the 13C NMR training protocol).
28. If you do not need a ^{13}C NMR, finish up by clicking on the 'Acquire' tab in the TopSpin Menu bar and then click the arrow on the 'Spin' icon and choose "turn sample rotation off"
29. Click on the arrow on the 'Sample' tab and choose "turn on sample lift air"
30. Remove your sample from the instrument and place the standard sample into the spinner. Use the depth gauge and then wipe the sample tube and spinner with a Kimwipe.
31. Place the standard sample into the NMR
32. Click on the arrow on the 'Sample' tab and choose "turn off sample lift air." Wait until the sample is lowered into the instrument and the lift air is no longer audible.
33. Click the 'Lock' icon and choose CDCl_3
34. Replace the magnet cap
35. Enter end time in logbook