

## <sup>13</sup>C NMR Training Protocol for Fourier 300

1. Follow procedure for collecting <sup>1</sup>H NMR through the print step, then collect <sup>13</sup>C NMR data (If you do not collect a <sup>1</sup>H NMR first, follow the **Sample Related Steps** in the <sup>1</sup>H NMR Training Protocol now)
2. **Record run in logbook**, if it is not done already
3. Drag the 13C folder from your research group's subdirectory (in the “**NMR Data Browser**” screen) to the “Bruker TopSpin 3.1” window
4. 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 C13CPD next to the Experiment section. Click OK once you have finished
5. Type "ns" into the lower left hand corner above the message box and press enter. Type the appropriate number of scans and press enter
6. Click the '**Acquire**' tab and click the '**Prosol**' icon and wait for the message box to read “**getprosol finished!**”
7. Click the '**Gain**' icon and wait for the message box to read "**Job succeeded**"
8. Type "ns" into the lower left hand corner above the message box and press enter. Type the appropriate number of scans and press enter.
9. Click the '**Go**' icon. Scanning will now begin. Wait until the message box reads "**Job succeeded**"
10. Click on the '**Process**' tab in the TopSpin Menu bar and click on the '**Proc. Spectrum**' icon
11. 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
12. If the phasing is off, click on the '**Adjust Phase**' icon and adjust accordingly. Click the '**return, save changes**' icon when finished
13. Click on the '**Integrate**' icon and highlight individual peaks or the entire spectrum as desired. Click the '**return, save changes**' icon when finished
14. 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
15. 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
16. 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.

- 17.** To finish, click on the '**Acquire**' **tab** in the TopSpin Menu bar and then click the arrow on the '**Spin**' **icon** and choose "**turn sample rotation off**"
- 18.** Click on the arrow on the '**Sample**' **tab** and choose "**turn on sample lift air**"
- 19.** 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.
- 20.** Place the standard sample into the NMR
- 21.** 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.
- 22.** Click the '**Lock**' **icon** and choose **CDCl<sub>3</sub>**
- 23.** Replace the **magnet cap**
- 24.** **Enter end time in logbook**