TopSpin 1.3 Acquisition Instructions

Revision – April 2016 for DPX 250 MHz (NMR-Thyme) and DPX 400 MHz (NMR-Paprika)

These are basic instructions setting up NMR experiments. Note: all commands are case sensitive! For more details contact the NMR manager. Never attempt an unfamiliar experiment without help!

1. Log in the computer with your chemistry domain account.
2. Double click on the TOPSPIN icon to launch the program.

CREATE DATA SET

3. Type **new** to create a filename for your new experiment. Enter a filename for your sample.
4. *Important:* Ideally your NMR data should be stored on one of the NMR network drives (found my clicking on My Computer). Set the data set owner to your username (same as log-in) and the disk unit (DU) to **X:** (ie, the letter of the appropriate network drive).

INSERT SAMPLE

5. Enter **bsmsdisp** in the XWIN-NMR command line.
6. Check that the [MAIN] tab is opened.
7. Click on [LIFT] to start the lift air.
8. Insert your sample when the lift air in on.
9. Click on [LIFT] to turn off the lift air.
10. Click on [SPIN] to spin your sample (it will turn green when spinning).

SELECT NMR EXPERIMENT

11. Type **rpar** and press enter. Select your desired experiment from the long list.
12. Click on the [copy all] button at the bottom of the panel.
13. *Important:* Type **gpro** after you click [copy all] to set the power levels and pulse widths, otherwise you will not see a signal.

LOCK AND SHIM

14. Open the lock display by typing the command **lockdisp** or double-click on lock window.
15. Read in the standard shim file which is posted for each instrument (type **rsh QNPtly, rsh BBOtly**, or **rsh BBItly**).
16. Type **lock** or click  on the command line and choose your solvent from the list. Wait until finished.
17. Open the [MAIN] panel of the BSMS Display.
18. Decrease the lock gain [GAIN] to move the lock signal within the top half of the grid. Adjust the value by clicking the [STEP -] button.
19. Click on the [Z] shim. Click the [STEP +] and [STEP -] button to adjust the shim value. Adjust the shim value until the lock signal shown in the Lock Display is at a maximum. If the lock signal reaches the top of the Lock Display, click on [Gain] (under the Lock subheading) and lower the signal with the [STEP+] and [STEP -] button.
20. Click on the [Z2] shim. Repeat as for the [Z] shim.
21. Alternate between the Z and Z2 until there is no improvement.
22. Minimize the BSMS display.

ACQUIRE SPECTRUM

23. Set the receiver gain and start the acquisition with the command **rga**;**zg**. The acquisition progress automatically will appear.
24. To save and view the data during the middle of the acquisition, enter **tr** to transfer your data to disk (optional).
25. Enter **efp** to process your saved data.

PROCESS SPECTRUM

26. To phase, enter **apk**.
27. To calibrate the spectrum, click on the calibrate icon.  Without clicking on another icon, using the left mouse button, click on the peak of interest and enter the chemical shift.
28. To integrate the spectrum, click on the integrate icon.  Then, click on  . Using the left mouse button, click and drag the regions of interest. Click  .
29. To pick peaks, click on the peak picking icon.  Without clicking on another icon, using the left mouse button, click and drag a box around the peaks of interest (the bottom of the box is the minimum threshold level). Click  .
30. To print, type **plot** and use the plot editor as usual.

FINSIHING UP

31. Type **new** to begin a new experiment on this sample or:
32. Stop the spinning with the [SPIN] button and turn off the lock using the [LOCK] button. Eject your sample with the [LIFT ON/OFF] button. Remove your sample, turn off the lift air [LIFT ON/OFF], exit TOPSPIN, and log off the computer.