

Running 1D Proton/Carbon NMR Experiments

Login to computer

Make sure nobody has logged into the computer

Using your group ID and password, login to the computer

TOPSPIN automatically starts once you login.

Sample preparation

Use clean and dry NMR tubes

Use 528-PP/535-PP NMR tubes for 500/600 MHz NMR

Always use a solution height of 5 cm (~ 0.5 ml)

Turn on LIFT ON button on the BSMS keyboard to remove the standard sample.

Place your sample in the spinner and use the depth gauge to adjust the sample depth

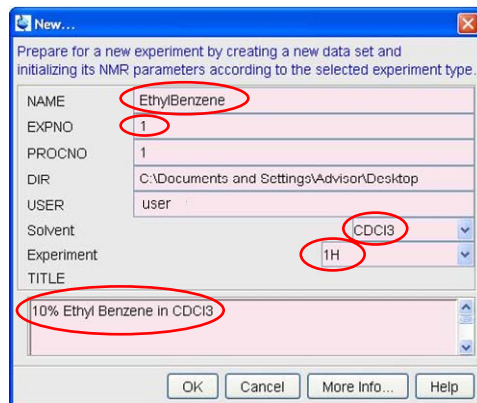
Wipe the sample tube clean before inserting into magnet


Make sure you hear the air flow before inserting the sample.


1D Proton/Carbon Experiment

Acquisition

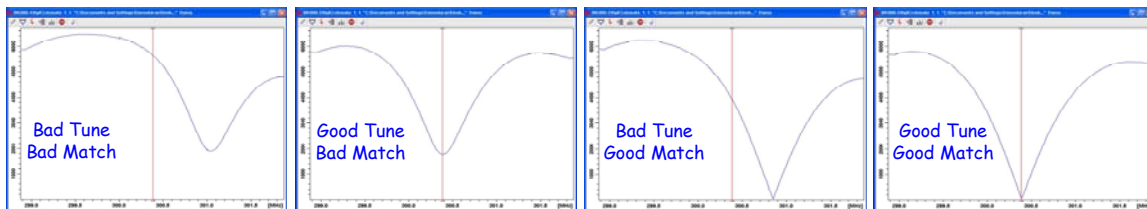
1. Type “new” on the TOPSPIN window command prompt.

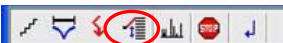



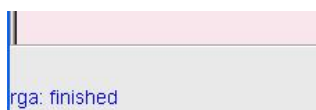
2. Type a name and experiment # (Start with 1) for your experiment
3. Choose your solvent
4. Choose “1H” for proton NMR: Choose “13C” for Carbon NMR
5. Set a title for your experiment and click
6. Type “rsh temp” to load a good shim file
7. Click on  to get the lock display

8. In the lock display window click on  and select your solvent
9. Type “**wobb**” to tune the probe.

Adjust Tune and Match rods to get “Good Tune & Good Match” as shown below.




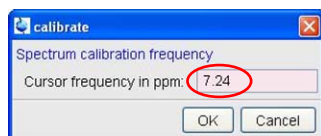
10. If your expt involves more than one nucleus, click  to tune the next channel.
11. Click  to stop wobb.
12. Shim Z1 and Z2 for the best homogeneity
13. On the TOPSPIN window type “**rga**” to set receiver gain.








14. Type “**zg**” - to start acquisition.

Processing

1. Type “**ef**” - to Fourier Transform the FID
2. Type “**apk**” - for automatic phase correction.
3. Type “**abs**” – for automatic baseline correction
4. If peaks are not at the right position, zoom the peak for calibration and click 
5. Click on the top of the peak and enter the right value in ppm



6. For Peak Picking, display the entire spectrum and type “**pps**”
7. For proton NMR integration click on 
8. Automatic integration is performed by abs. Click  to select all integrals and then click  to delete them.
9. In the integration menu bar click on 

10. Set the cursor line, starting at the left of the spectrum to the left of the first peak to be integrated, click the left mouse button and drag the cursor line to the right of the peak, then release the mouse button
11. Repeat step 10 for the remainder of the peaks
12. Click  to save the integrals and return from the integration menu

Plotting

Simple Plotting

Use Ctrl-P or Go to File -> Print

Advanced Plotting


Type “**plot**” in the TOPSPIN window and use the Plot Editor.

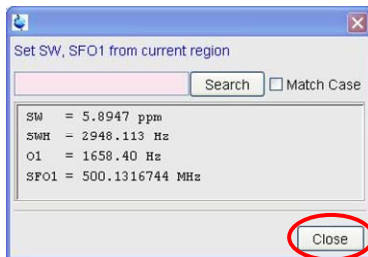


After finishing your experiments

Running 2D NMR Experiments

Preparation for 2D

1. Run a normal ^1H NMR experiment before running any 2D experiment. Follow the above procedure.
2. Expand the region of interest in the ^1H NMR spectrum. Then click 
3. When the following window comes up, note down the “SW” and “O1” values

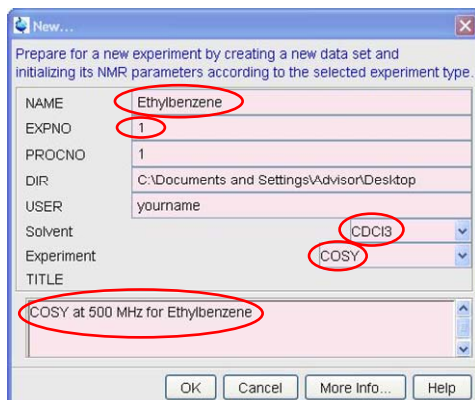


4. Click on “Close”
5. After referencing the ^1H spectrum, type “sr” and note down the exact value.
6. Stop sample spinning
7. Shim Z1 and Z2 iteratively till there is no further change. Now shim X and Y iteratively till there is no further change. Go back to Z1 and Z2 till there is no further improvement.

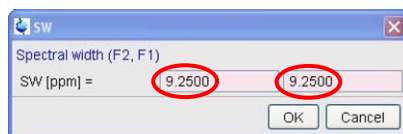
^1H - ^1H COSY

Acquisition

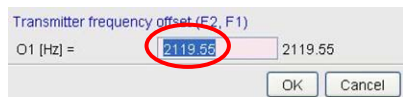
1. Type “new” on the TOPSPIN window command prompt, to create a new data set.




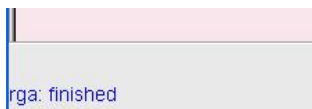
- Type "**sw**" and enter the value that you had noted down from the ^1H NMR spectrum into both F1 and F2 dimension



- Type "**o1**" and enter the value that you had noted down from the ^1H NMR spectrum.



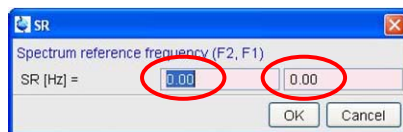
- Check 1H tuning and matching.
- Set the required number of scans by typing "**ns**"
- You can check the time required for finishing the experiment by clicking 
- Type "**rga**" to set receiver gain.



- Type "**zg**" - to start acquisition.

Processing

- Type "**sr**" and enter the exact value noted down from the 1H spectrum.



- Type "**xfb**" - Fourier Transform in both the dimensions.
- Type "**abs2**" - automatic baseline correction in the f2 dimension.
- Type "**abs1**" - for automatic baseline correction in the f1 dimension.
- Type "**sym**" - makes the 2D spectrum symmetric.

Plotting

Simple Plotting

Use Ctrl-P (or) Go to File -> Print

If you want to have external ^1H projection, right click on the projection spectrum region and choose the external projection file (1H data)

Advanced Plotting

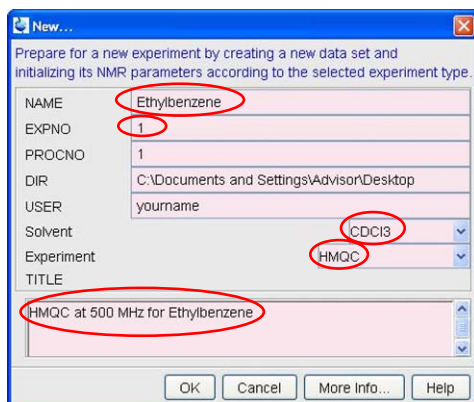
Type "**plot**" in the TOPSPIN window and use the Plot Editor.



After finishing your experiments

$^1\text{H} - ^{13}\text{C}$ HMQCAcquisition

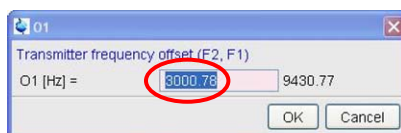
1. Type “**new**” on the TOPSPIN window command prompt, to create a new data set.




2. Type “**sw**” and enter the value that you had noted down from the ^1H NMR spectrum into only the F2 dimension (first box)



3. Type “**o1**” and enter the value that you had noted down from the ^1H NMR spectrum.



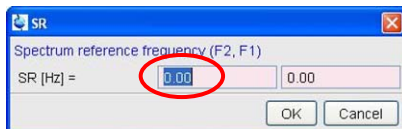
4. Check ^{13}C and ^1H tuning and matching.
5. Set the required number of scans by typing “**ns**”
6. You can check the time required for finishing the experiment by clicking 
7. Type “**rga**” to set receiver gain.



8. Type “**zg**” - to start acquisition.

Processing

9. Type “**sr**” and enter the exact value noted down from the ^1H spectrum, only for the F2 dimension.



10. Type “**xfb**” - Fourier Transform in both the dimensions.
11. Type “**abs2**” - automatic baseline correction in the f2 dimension.
12. Type “**abs1**” - for automatic baseline correction in the f1 dimension.

Plotting

Simple Plotting

Use Ctrl-P (or) Go to File -> Print

If you want to have external ^1H projection, right click on the projection spectrum region and choose the external projection file (1H data)

Advanced Plotting

Type “**plot**” in the TOPSPIN window and use the Plot Editor.

