Acquisition and Processing of a 1-D & 2D NMR Experiment

NMR Access information

User name:
Password:

Safety First: Make sure you read NMR safety related information on the NMR website: http://nmr.uwinnipeg.ca

Write your name and other required information in the log book.

Experiment Setup

Start

1. Click on the ‘Start’ tab in the TopSpin Menu bar and ‘Create Dataset’ from the menu below it. Enter the required information into the new dataset window.

2. Use the tab “Read Pars.” to select the type of 1D or 2D experiment from the list on the pop-up window. If the required experiment is not listed, change the Class = HighRes to any. You will be able to find the experimental parameters you need. If not, then contact me :) Phone: 9876 or email r.vakili@uwinnipeg.ca
Acquire

1. Click on the ‘Acquire’ tab in the TopSpin menu bar and ‘Sample’ from the menu below it. Select ‘ej’ by clicking on it. Wait until the sample lift air is turned on and remove any sample at the top which may have been in the magnet. Place the sample in the holder in to the lift air at the top of the magnet. Select ‘ij’ by clicking on it. Wait until the sample is lowered down in to the probe and the lift air turns off. A clicking sound will be heard and the “?” icon from the Sample area will disappear.
2. Click on ‘Lock’ and Select ‘90%H2O and 10%D2O’ by clicking on it. Note the lock signal in Lock Display menu. Once sample is locked you see a straight line.

3. Perform a Tune (automatic tuning) by clicking on ‘Tune’ icon on the menu. Wait until the tune is complete.

4. Using the ‘AcquPars’ tab make the required changes to the acquisition parameters:

   **Under experiment:**
   - Set DS= 2 (this is the number of dummy scans)
   - Set NS= desired number (this is the number of actual scans)

   Make other changes as required.

5. You can turn “Spin” On/Off as needed.
6. Press Shim, this will execute “Topshim”. Wait until the operation is complete.

Some sample may require manual shimming. By double clicking on Sample icon in the lower tool bar you can bring up the BSMS Control Suite and make the require adjustment. This is covered during training.

You most likely need to adjust the Z, Z2 & Z3. Do not adjust other shim parameters.

7. Select ‘Prosol’ by clicking on it. This will load the pulse width and power levels in to the parameter set.

8. Select ‘Gain’ by clicking on it.
Select ‘Go’ by clicking on it. Wait until all FID collected base on the number of scans “NS”.

**Process**

1. Click on the ‘Process’ tab in the TopSpin Menu bar.

NOTE: This executes a processing program including commands such as an exponential window function ‘em’, Fourier transformation ‘ft’, an automatic phase correction ‘apk’ and a baseline correction ‘abs’. Other options are available by clicking on the down arrow inside the ‘Proc. Spectrum’ button.

2. Click on the ‘Proc Spectrum’. Your collected FID will be processed.
3. Calibrate your spectrum by clicking on ‘Calib. Axis’. Place your cursor on the highest point of the water peak and reference it as 4.63 ppm. You can expand your peaks by left clicking before the peak and holding the click then drag the cursor over to the other side of the peak.
4. You can use ‘Pick Peaks’ by clicking on it and using your cursor to identify the peak(s) by clicking on it.

Remember to ‘save and return’ after you are done. You can not preform any other action before closing the sub menu.

5. You can use ‘Integrate’ by clicking on it. Using your cursor to integrate the peaks. Remember to ‘save and return’ after you are done. You can not preform any other action before closing the sub menu.
6. Plot your spectrum by Clicking on the ‘Plot’ tab and using the blue arrow, you can go to home area and add parameters, title, more spectrum, etc. You can expend your peaks by selecting the spectrum in the plot and clicking on the ‘Expend’ button in the home menu and using your cursor to select your peak. Once done, click on printer icon and print a copy of your spectrum.

Publish

Under the “Publish” tab you will be able to print and also export your results as a PDF. Hope one day we can email out the results.
2D NMR

Create a new data set as per this guide. In read Parameter select the desire 2D experiment. Make sure you re tune if required. Other operations are as described above. Just note that if you running COSY or other correlation spectroscopy, you need to Symmetrize your spectrum.

Need more help?

Click on the “?” icon. Manuals are available to you for bed time reading. Remember, you can always ask for help. With NMR system, If you are not sure just ask . Phone: 9876

email: r.vakili@uwinnipeg.ca

Before you go

Remove your sample from the NMR magnet as stated in part 2 of “Experiment Setup”. Logout and record the time in the log book.

You are done. Make sure you clean the area and that the door to the NMR facility is closed after leaving the room.