Routine ^{1}H and ^{13}C NMR Data Acquisition
Basic Topspin Processing Guide
For PSC and NSC Bruker 400 NMR Spectrometer

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LC=Left click; DC=double left click
Black and bold command need to be typed and followed with enter.

PSC Data Directory: C:\Bruker\TopspinX.X\data\nmr\'userid'
NSC Data Directory: /opt/topspin2.1/data/'userid'

Step	Command	Notes
1	Login to the computer	**DO NOT** share your user ID with unauthorized person.
2	Start Topspin	PSC: Use Topspin3.1 only; NSC: Use Topspin2.1.
3	**Open an old spectrum and type 'new'**	*Open Dataset window.* Change *dir* or *exp* or both. You need a new page for NMR data. Type in a title area your note for the experiment
4	**bsmsdisp** <etr>	Unlock (make sure lock/on-off button is not highlighted) Unspin (make sure spin button is not highlighted).
5	**ej** <etr>	*This will eject sample from the magnet.* Please remove spinner turbine from the top of the magnet without leaning on the magnet; Take out D_2O sample from the spinner turbine; Position your sample using the sample depth gauge; Make sure the eject air is still ON (should hear the sound of the air), place the turbine with your sample on top of the magnet, if there is enough support, release the sample;
6	**ij** <etr>	*Insert sample into the magnet.* Double check that the sample status on the bsmsdisp window showing **'down'** . **Close BSMSDISP display window**.
7	**rpar** <etr>	Read the appropriate parameter set for expt. *For proton: rpar 'Proton';* *For Carbon in PSC, rpar "C13CPD';* *For Carbon in NSC, rpar C13CPDduz from user menu.*
8	**lockdisp** <etr>	*Lock display window opens up*
9	**rsh** <etr>	s*him file list opens up.* LC on the file "lastbestbbo" or some other files similar.
10	**lock** <etr>	LC on the appropriate lock solvent. Wait until "locking:finished" message appears
11	***atma*** *<etr>*	*Automatic probe tuning and match starts.* *Wait till "atma:finished" message appears. For 13C experiments, please wait till both carbon and proton channels are done.*

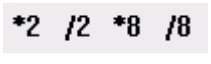
12	**bsmsdisp** <etr>	*Open BSMS board for manual shimming. Tune on Z, Z2, and Z with step size of 5,10, and 5 to maximize the lock signal. Adjust gain if necessary and close BSMSDISP afterwards.*
13	***topshim 2h***<*etr*>	*Automatic shimming starts to optimize for 2H.* *Wait till the "topshim completed" message appears.*
14	**ased** <etr>	AquProc setup mode.
15	**getprosol** <etr>	*Read corresponding probe parameters.*
16	**rga** <etr>	*Automatic receiver gain optimization starts.* Wait till the "rga:finished" message appear
17	**expt** <etr>	Calculate the time to finish the exp; Adjust "ns" if the acquisition time is too long; DO NOT EAT other user's time.
18	**zg** <etr>	*Start data acquisition.* Wait till the "acquisition:finished" message to appear.
19	**efp** <etr> **apk** <etr> **abs** <etr> or command '**duzproc**' does all three together.	Simple data processing If additional experiments are required for this sample, repe step 3, 7, (11),14,15,16,17,18. Type *tr* to transfer data for processing if exp not finished; Type *halt* to stop experiment and save data; **STOP ALL UNFINISHED EXP BEFORE TO STEP 20** **If doing 13C, type lb=5 before 'duzproc' to increase S/N**
20	**bsmsdisp** <etr>	Unlock (make sure lock/on-off button is not highlighted Unspin (make sure spin button is not highlighted.
21	**ej** <etr>	Remove the spinner turbine from the top of the magnet. Take out your sample from the spinner turbine. Position the D2O sample using the sample depth gauge; Make sure the eject air is still ON (should hear the sound of the air), place the turbine with with D2O on top of the magnet, if there is enough support, release the sample;
22	**ij** <etr>	*the D2O sample is inserted into the magnet.* Wait until the sample status on the bsmsdisp window showing 'down'. Then close BSMSDISP display window.
23	**lock d2o** <etr>	Locks d2o sample. Wait until "locking:finished" message appears
24	**Transfer your data**	Please process this immediately. Data older than two weeks may be deleted without notice.
25	**exit** <etr>	**Always type 'exit' to terminate the program.** Please **DO NOT** just click the crossbar as it does not always terminate all active commands. Log out PC. **Fill out the log book** Record all error messages from the PC screen to logbook if instruments are malfunctioning. **Email or call Dr.Du if the status is not ok.** Dr.Zhenming Du

TOPSPIN Processing Guide

Dummy Data Processing guide

tr: transfer unfinished 1D fid;
efp: em+ft+ph
apk: automatic phase correction
abs: automatic baseline correction

Detailed Data Processing guide

1. Opening Files and FTing Data
    a. Click on File -> Open and find your file name.
    b. Click OK. If multiple experiments were run with that filename, you may have to select an EXPNO.
    c. Type "**efp**" to FT your data.
2. Zooming In / Zooming Out
    a. 

Increase/decrease vertical scale by factor of either 2 or 8.
    b.  Press LMB down while dragging mouse up/down to inc/dec vertical scale manually.
    c.  Scales to tallest peak.
    d.  Scales out full sweep width.
    e.  Moves spectrum upfield/downfield.
    f.  Moves spectrum up/down.
    g.  Switches scale between Hz and ppm.
3. Zooming In / Out Using the Mouse
4. Manual Phasing
    a. Click on  to enter phase mode.
    b. The buttons above your spectrum should look like this:
    

    c. Click and hold the LMB on the 

button to perform zero-order phasing.
    d. Click and hold the LMB on the

button to perform first-order phasing.

e. Click on  to save and return when you are done phasing.

5. Calibrating the Spectrum

   a. Click on  .

   b. Click on your reference peak (solvent or TMS).

   c. Enter the chemical shift of that peak.

6. Integration

   a. NOTE: It is easiest to zoom in on a small region and use the  buttons to move around while integrating!

   b. Click on the  button to enter integrals mode. The toolbar above your spectrum should change to this:

   c. Click on  . The box should turn green, indicating you are now integrating.

   d. <u>Click and hold</u> with the LMB on the left side of the peak you want to integrate, and drag <u>while holding down</u> LMB to the right side, then let go when you are done integrating.

   e. Repeat step d for each peak or region of interest.

   f. To adjust bias and slope, click and hold LMB on  while dragging the mouse.

   g. To Open/Save a region file:

      1. Click on  to open or save a region file already associated with that spectrum.

   h. To Delete an integral:

      1. Right click on the region you want to delete and choose “Select”. Once selected, the integral region should turn yellow.
      2. You can select many regions at once by repeating step 1.
      3. Click on  to delete the selected regions.

   i. When you are done, click  to save and return the integral file.

7. Peak Picking

   a. Click on  to enter peak picking mode. The toolbar above your spectrum should change to look like this:

b. Place your cursor in the upper left hand corner of the spectrum.

c. Click and drag LMB down to the lower right hand corner of the spectrum. d. The maximum and minimum are automatically set based on the region you select.

e. To delete your peak picking regions, click  . f. Click on  to save and return.

8. Manual Peak Picking

a. Click on  (green) to manually select peaks. A red cursor will appear. b. Click the LMB on each peak you want to pick.

c. Click  to save and return.

9. Plotting

a. Type ‘plot’.

b. A default layout of the 1-D spectrum appears.

c. To adjust the horizontal scale, click the spectrum area.

1. Select ‘Edit’.
2. Change the x-axis to be whatever range you need (ie 10 – 0 ppm)
3. Click Apply and OK when you are done;

d. To adjust the vertical scale, move the crosshair into the spectrum area.

1. Select ‘1D/2D-Edit’.
2. Use the buttons to adjust the vertical scale of the spectrum.
3. Click Apply and OK.

e. Click on ‘File’.

f. Click on ‘Print’.