

Bruker DPX 400MHz Nuclear Magnetic Resonance (NMR) Spectrometer

Instrument instructions can be found at:

<http://academic.bowdoin.edu/chemistry/resources/instructions.shtml>

If you have any problems with the instrument or would like training, please contact

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1. Startup Procedure

- a. **Login to computer.**
 - i. At the login screen, select your username and hit Enter.
 - ii. Type your password and hit Enter.
- b. **Open TopSpin 1.3.**
 - i. Double click on the Top Spin 1.3 icon on the desktop.

2. File Browser Overview—Review Before Creating New File

- a. **A standard Bruker dataset is a directory tree, not a single file.**
 - i. `<dir>/data/<user>/nmr/<dataset name>/<expno>/pdata/<procno>`
 - ii. The information in “< >” is what you enter in the “New...” window that appears when you type “edc”
 - iii. Example:
 1. Name (<dataset name>): **proton**
 2. Expno (<expno>): **1**
 3. Procno (<procno>): **1**
 4. Dir (<dir>): **/opt/topspin/chem226**
 5. User (<user>): **Orgolab**
 6. Directory created will be - **/opt/topspin/
/chem226/data/orgolab/nmr/proton/2/pdata/1**
- b. **To open a data file, navigate to the “expno”.** Click and hold the left mouse button on the folder icon just to the left of the “expno” number. Drag the folder to the spectrum window.

3. Creating a Data File

- a. **Type edc in the command line and hit Enter.**
- b. **A “New...” window will open.**
- c. **Type experiment name in the “Name” field.**
- d. **Type 1 in the “Expno” field.**

Note: To save subsequent experiments within the same experiment name, type edc and hit Enter. Leave the Name field untouched but increase the Expno field by one.

Note: To switch between experiments with the same experiment name, in the command line type re exp# and hit Enter (i.e. re 1 or re 2).

- e. **Type 1 in the “Procno” field.**
- f. **The “Dir” field is the path where data will be stored.** The path is “/opt/topspin/username”. Where “username” is, type the name of the account you logged in as.
- g. **Type your name in the “User” field.**
- h. **Select Solvent Type from the drop-down box.**
- i. **Select Experiment from the drop-down box.**
- j. **In the “Title” box, type the name you want to appear on your spectrum.**
- k. **When you are finished, click OK.**

4. Selecting a Parameter File

Note: If you just created a new file following the previous directions then it will have exactly the same parameters as the file TopSpin first opened to. If it was a proton file, you have created a new proton file and you can use it without further modification (if you want to collect a proton spectrum). If you do not know what kind of file it was, it doesn't matter as you can call up the parameters you want.

- a. **For proton (^1H) experiment:**
 - i. Type “rpar 1h226” in the command line and hit Enter.
 - ii. A “rpar” window will open.
 - iii. Make sure all four fields are highlighted and click OK.
- b. **For carbon (^{13}C) experiment:**
 - i. Type “rpar bc13c” in the command line and hit Enter.
 - ii. A “rpar” window will open.
 - iii. Make sure all four fields are highlighted and click OK.

5. Creating an Experiment Title

- a. **Type “setti” in the command line and hit Enter.**
- b. **The “Title” tab will become active.**
- c. **Place the cursor in the box and type your title.**
- d. **Type “.sret” in the command line and hit Enter.**

6. Insert your Sample

Note: Use the ladder if you cannot comfortably reach the top of the magnet – please do not lean on the magnet, it is top heavy.

- a. **Clean your sample NMR tube and spinner with a dry Kim-Wipe.**
 - i. Throw the Kim-Wipe away.
 - ii. Remove any labels from your NMR tube.
- b. **Set sample NMR tube to the correct height (VERY IMPORTANT) in the spinner to enable good shimming.**

Note: Occasionally a sample tube will break when it is inserted or removed from the spinner (sample holder). This is not a big deal if you simply get Rick or Celeste to remove the broken tube from the spinner. DO NOT try to do anything with the spinner if the tube breaks in it, as it is easy to damage the expensive (\$250) spinner.

- c. **On the magnet, remove the dust cap.**
- d. **On the BSMS unit, press the “LIFT ON/OFF” button (“LIFT ON/OFF” light will illuminate green when ON)-wait to hear the air purge before holding sample over the magnet!**
- e. **After a few seconds, an air purge will come on.**
- f. **Holding the sample (not the spinner), carefully place the sample in the magnet.**
- g. **On the BSMS unit, press the “LIFT ON/OFF” button (“LIFT ON/OFF” light will NOT be green when air purge is OFF).**
- h. **After a few seconds the air purge will lower the sample into the heart of the magnet.**
- i. **Replace the dust cap on the magnet.**
- j. **On the BSMS unit, press the “SPIN ON/OFF” button (“SPIN ON/OFF” light will illuminate green when ON).**

7. Locking the Sample

- a. **Type “lock” in the command line and hit Enter.**
- b. **A “lock” window will open.** If the TopSpin window is maximized, the Lock window will be pushed behind it. You can bring it forward again by clicking on the appropriate window in the taskbar (bottom of screen) or click on the orange[3]square in top right corner of screen.
- c. **Select solvent used to prepare sample and click OK.**
 - i. The instrument will automatically adjust the necessary parameters and lock the sample.

8. Shimming the Sample

- a. **Make sure “Fine” button is on** (“Fine” light on BSMS unit will illuminate green when ON).

- b. **To shim the magnet, you will adjust Z¹ and Z².**
- Press the “Z¹” button (“Z¹” light will turn on) and rotate the wheel, in either direction necessary, until the signal improves (lock signal in “lock” window moves up).
 - Adjust Z¹ until the signal is maximized.
 - Press the “Z²” button and rotate the wheel.
 - Alternate between Z¹ and Z² until no further improvement is obtained.

Note: If the signal level is maxed out (the signal is at the top of the “lockdisp” window), you will need to lower the lock gain. Press the “LOCK GAIN” button (“LOCK GAIN” light will turn on) and lower the value (turn the wheel counterclockwise) until the signal is about 1-2 inches below the top of the “lockdisp” window.

- c. **Press the “STDBY” (standby) button (“STDBY” light will illuminate green when ON), when finished.**

9. Acquiring the Spectrum

- Initialize instrument.**
 - Type “ii” in the command line and hit Enter. ***Note***-give it time to complete each step before typing the next command.
- Set receiver gain (for Proton NMR only).**
 - Type “rga” in the command line and hit Enter.
- Start acquisition.**
 - Type “zg” in the command line and hit Enter.

10. Processing the Data After Acquisition is Complete

- Fourier transform the time domain data to a frequency domain.**
 - Type “ft” in the command line and hit Enter.
- Autophase spectrum.**
 - Type “apk” in the command line and hit Enter.
- Correct baseline.**
 - Type “abs” in the command line and hit Enter.
- Set reference peak.**
 - Type “.cal” in the command line and hit Enter.
 - Move the red line cursor to the top of the reference peak, then click the left mouse button.
 - In the popup window, type in the reference chemical shift value and hit Enter.
- Integrate spectrum.**
 - Type “.int” in the command line and hit Enter.

- ii. Click on the second icon from the left (“Define new region using cursor (toggle”). To integrate a peak, move the red cursor line to where you want the integration to start and click and hold the left mouse button. While holding the left mouse button, move the second red cursor line that appears to where you want the integral to end. Release the left mouse button when finished.
 - iii. To phase an integral, you must first select it. Move the red line cursor over the integral and click the right mouse button. A shortcut menu will appear. Select “Select/Deselect”. Near the bottom of the screen where the value of the integral is listed, a yellow bar will appear. You can select more than one integral, but it is not recommended when phasing. There are two icons (Interactive bias correction and Interactive slope correction) which are used to phase the integral. When finished, move the red line cursor over the integral and click the right mouse button. On the menu that appears, select “Select/Deselect”. Repeat this step until all the peaks are phased.
 - iv. There are two options when assigning a value to your integrals. You can normalize all the integrals to a value, or you can assign an integral a value and the remaining integrals will be adjusted accordingly. To normalize, click the right mouse button when the red cursor line is over an integral and select “Normalize”. Enter the value you would like the sum of all the integrals to be and click OK. If you would like to calibrate one integral, move the red line cursor over the integral and click the right mouse button. Select “Calibrate”, enter a value and click OK. It is not necessary to select an integral before calibrating it.
 - v. When finished, type “.sret” in the command line and hit Enter.
- f. **Peak picking the spectrum.**
- i. Type “.pp” in the command line and hit Enter.
 - ii. Define the peaks you want included by drawing a box around the peak(s). Move the pointer cursor (attached to the red line cursor) to the top left corner of where you want the box to start. Click and hold the left mouse button and begin drawing a box around the peaks. The box will be highlighted green. Release the left mouse button when the box is complete. If there are peaks in another region that you would like labeled, draw another box in that region. Delete regions by moving the red line cursor over the box and clicking the right mouse button and selecting “Delete Region Under Cursor”.
 - iii. To view list of peaks, click the right mouse button (anywhere on the screen) and select “Show Peak List”.
 - iv. When finished, type “.sret” in the command line and hit Enter.

11. Printing the Spectrum (standard layout)

Note: If you type “plot” instead of going to File > Print, the X & Y limits Plot Editor will use will be based on how they are set on the screen, not how they are saved in the template.

- a. **Go to File > Print.**

- b. A **“Print [Ctrl+P] – prnt” window will pop up with two sections.**
 - i. Options
 1. **Select “Print active window” and click OK to print the spectrum as is in the window. (Skip to step c. if using this option)**
 2. Select “Print with layout – plot directly [autoplot]”, using a specified template. (see required parameters below)
 - ii. Required parameters
 1. To use a saved template, set “Layout=” to the template name.
 2. For a generic 11 x 17 layout, set “Layout=” to “+/Bowdoin.xwp”.
 3. For a generic 8 ½ x 11 layout, set “Layout=” to “+/Bowdoin8x11.xwp.”
- Note: When selecting plot limits, you need to determine if you want the X & Y limits to be the same as they are on the screen or how they are saved in plot editor.
4. In “Use plot limits”, select “from screen/CY” or “as saved in Plot Editor”.
 5. Click OK.
- c. **Click “Print” and then click “OK” in the page setup. The spectrum will print on the inkjet color printer (Carbon) located in Rm. 38 near the prep room.**

12. Printing the Spectrum (custom layout)

Note: After creating and saving a template, use Step 11 to print out spectra using the template you create.

- a. **To create a plot layout, type plot in the command line and hit Enter. The Plot Editor will open.**
- b. **Select paper size – this should be the same size as you selected in Step 12.**
 - i. Go to File > Print.
 - ii. Click on the Setup button.
 - iii. Select either “11 x 17” or “letter” (letter is 8 ½” x 11”).
- c. **Basic Plot Editor commands.**
 - i. Add a new spectrum window.
 1. Click on the “Create 1D spectrum” icon. Once selected, move cursor over to layout window and click and hold the left mouse button while you draw a box. This box will be your new spectrum window.
 2. When you are finished, click on the “Mark objects”.
 - ii. Add a title box – Click on the “Create Title” icon. Once selected, move the cursor over to the layout window and click and hold the left mouse button while you draw a box.
 - iii. Change color of the objects – Click on the “Mark Objects” icon and select a window (eight green boxes will appear around a selected window). Near the top of the screen, click on the Edit button. An “Edit Display Object” window will open up. Scroll down to the “1D Spectrum Object Editor” section. At the bottom of this section will be attributes with a Peaks, Integral, and Scaling Info button. Clicking one of these button will pull up another window that will let you change the color of the a particular feature.
 - iv. Adjust the appearance of the spectrum inside the window - Click on the “Mark Objects” icon and select a window (eight green boxes will appear around a

selected window). Near the top of the screen, click on the 1D/2D-Edit button. At the very top choose the scope. Adjustments will only affect what is selected (dark gray color). The top row of icon will move and zoom the spectrum. The second row will increase/decrease the height of the spectrum. The third row of icons is used to return the spectrum to its original scale. Below the icons are more check boxes that shows what will appear on the spectrum. If a feature is selected, it will be shown on the graph. Options here include X-Grid, Y-Grid, Show Peaks, and Show Integrals.

- v. To remove an object, click on the “Mark Objects” icon and select a window. Near the top of the screen, click on the Delete button. The window will be removed.
- vi. Mark Objects – used to resize and move objects on the screen.
 - 1. To move, click and hold the center wheel on the mouse. This will grab the object. Move to the new location and release the center wheel.
 - 2. To resize an object, left click once on the object and eight green boxes will appear around the object. Move cursor over a green box until it changes into an arrow cursor. Click and hold the left mouse button and move the mouse to resize.
- d. **Save template.**
 - i. In the “Selection” box, navigate to “\opt\topspin\plot\layouts\”. You do this by double clicking the folders listed vertically in the left column.
 - ii. Type the name you want for the template after this directory.
 - 1. Example: “\opt\topspin\plot\layouts\myplot.xwp”

13. Finishing Up

- a. **To remove your sample from the NMR.**
 - i. **Take the dust cover off the top of magnet.**
 - ii. **Press the Spin on/off button on BSMS unit—green light will go off.**
 - iii. **Press the Lift on/off to turn ON the air purge—green light will illuminate.**
 - iv. **Sample will pop up and you can remove the sample.**
 - v. **Press the Lift on/off to turn OFF the air purge—green light will go off.**
- b. **Carefully slide the spinner off the sample and place the spinner in the hood.**
- c. **Click the Quit button to close “lockdisp” window, or “X” out the lockdisp window.**
- d. **Close the TopSpin program (File > Exit), or “X” out of the program.**
- e. **Click OK when the “Close TopSpin” window pops up.**
- f. **Click on “System” in menu bar and scroll to “Logout Orgolab”, or click on the “door” icon in the menu bar to log out of the current session and click “Logout”.**
- g. **Be sure to clean up the area for the next user.**