Bruker NMR Topspin Manual

For the Georgia Tech NMR Center

Topspin is an icon driven software package. This means that instead of reading words and typing commands in the software, most of the things will be carried out by clicking icons on the screen. If you have used Bruker software where you typed in commands these will still work If you are not sure what an icon does pass the mouse over an icon and you will be given a written hint.

Starting the Bruker NMR software

After you log into the computer click on the **Topspin** icon.

Setting up the Experiment on all other Bruker instruments

Before you start collecting any data it is imperative that you create a new file to contain your data. In the START menu ***create new*** (you could also type ***new*** or ***edc*** on the command line). A window will open into which you should type a new NAME (filename) enter an EXPNO and PROCNUM (numbers only). If you wish to change the type of experiment you should do this now. Check the experiment directory and pick the experiment type from the lists supplied. You may see a very long list of experiments. To help finding the experiment you should check the box at the top that says **show recommended**. If you wish to make an exact copy of the current parameters click the button to use the current parameters. This will create a file containing exactly the same parameters as the experiment that you were in when you started to create the new file. Continue down the list filling in the important information. Be sure to click the radio button next to ***execute getprosol.*** Click ***save*** when you are finished.

Check to see that you have the correct parameters by clicking the AcquPar tab at the top of the spectrum window. Click nucleus in the left menu to be sure that you have the correct nucleus listed. If these are the proper parameters than you can continue, however, if you wish to change the type of experiment without changing the name and other parameters you must call up a new parameter set. This can be done by typing ***rpar***. The list of parameter sets will then appear. After finding the correct experiment click on it and then click ***copy all***.

The parameters that were picked can now be modified if necessary. Parameters can be entered in three different ways. If you do not know the parameters acronym ie. ns, d1, etc. you can go to the specific parameter tab at the top of the spectral window AcquPar (acquisitions); ProcPar, (processing) find it there and change it by clicking on the box next to the aronym and putting in the new value. If you know the acronym for the parameter that you want to change but wish to see what the current value it is before you change it just type the parameter acronym and a return. A box will then open containing the value for that parameter. You can then change the value or leave it unchanged. The easiest way is to type in the parameter acronym, a space and the new value ie. ***ns 16***.

Putting your sample in the instrument

All solution instruments except the Bruker 800 (Luke) and the Bruker 700 (Rey) which have auto-samplers always have a standard sample in the magnet. It is imperative that you remove that sample before you put your sample in the magnet.

To eject or insert your sample type ***e***j or ***ij*** respectively. When the standard sample is ejected from the instrument replace it with your sample that has been set to the correct depth with the depth gauge. Once the sample is down check the spin icon at the bottom of the window to see if your sample is spinning. Type ***ro on*** or ***ro off*** to start or stop the rotation (spinning) respectively.

Auto-Sampler Bruker 800 and 700 instruments
In order use the auto sampler place the spinner containing the sample, set to the correct depth, in the auto-sampler rack. If you put your sample under the lift mechanism you can type ***ij*** for insert or ***ej*** for eject. If you put the sample in a different position type ***sx xx*** where ***xx*** is the position of the sample. Always eject your last sample with ***ej***

Locking and shimming

Open the lock display window by clicking on the lock display icon or by typing ***lockdisp***.
To lock the sample type ***lock*** which will bring up a list of the known solvents. You can then click on the appropriate solvent name. If you know what the solvent name is in the software you can just type ***lock*** followed by the solvent name and the instrument will then automatically lock the solvent. The success of this procedure can be determined by looking to see if the line in the lock window is now about half way up the screen. If it is not you may want to bring up a good set of stored shims. This can be done by typing ***rsh*** which will cause a list of stored shims to appear. Click on a shim set for the installed probe that has the most recent date and then just repeat the locking procedure.

Gradient shimming

All of the Bruker solutions instruments have automatic gradient shimming. This can be started by typing ***topshim*** or clicking on the shim button in the **Aquire** menu

Tuning the probe:

Automatic Probe (ATM) Tuning

The probes in all of the Bruker instruments do automatic probe tuning. To start this process typing ***atma*** or clicking on the **Tune** button in the Aquire menu.

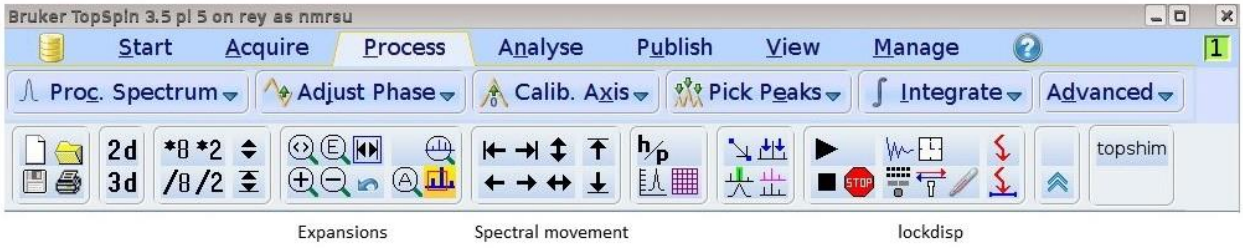
Collecting and Processing the 1D Data (for all systems)

Before you can collect the data you must set the receiver gain for an optimum value. This is done by typing ***rga***. When this is finished you can start the data collection by typing ***zg***.

If you wish to see the spectrum before the experiment is finish you can type ***tr*** to transfer the data from the console computer to the desktop for Fourier transform. If you wish to stop the experiment you can type ***stop***. If you do type stop before your experiment is completed than all data remaining in the console computer will be discarded causing you to lose it all, or any of your remaining data. If you wish to look at your spectrum or stop the experiment but keep the data up to that point you must type ***tr*** to transfer the data before typing stop. An alternative is to type ***halt*** which will complete the current scan, transfer the data and then do a stop.

Data Processing

When the data has been collected it must be transformed by typing ***efp***. This adds a line broadening factor, Fourier transforms the data and then adds some phase parameters. You should then see your spectrum on the screen. The spectrum can be manipulated with the icons on the top panel.



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Phasing

Automatic phasing is done by typing ***apk***. If the phase is still bad click the Adjust Phase tab in the menu which will open a menu bar. Put the mouse on **0** in the menu bar, hold down the left mouse button and move the mouse up and down to fix the phase of the tallest peak in the spectrum.

When this peak is correctly phased, move the mouse to the **1** and adjust the phase of the other peaks. When you are finished click the save and return icon.

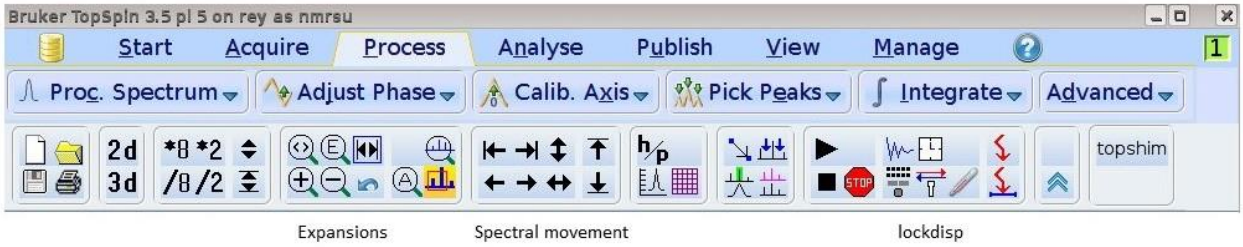
Spectrum Calibration

It is imperative that you calibrate your spectrum so that you can accurately compare chemical shifts. If you have TMS or other zero calibration substance you can type ***sref*** , which will automatically set the zero point. If you do not have a zero substance you will need to calibrate to the residual solvent peak. To do this expand the window around the solvent peak. Then in the process tab above select Calib.Axis to get a window where you can calibrate your spectrum. Put the curser directly through the peak that you want to calibrate and click the left mouse button. A window will appear where you can type in the reference location.

Baseline Correction

Sometimes the baseline is not flat or is not located or at the zero position which can affect the integration. If you wish to fix this problem type ***absn*** to automatically correct it.

Spectral Manipulations



Expansions:

You can expand the spectrum on the screen by moving the cursor to where you wish to start your expansion then hold the left mouse button. Drag the cursor across the screen and let go at the point where you want to end the expansion. You can also use the icons in the menu above.

Integration:

Click the **integrate** tab from the top Process menu bar to open a new integration menu. Be sure that the integration icon (just to the right of the yellow icon) is gold. If it is not then you will be

doing expansion and not integration. Pick the areas that you want to integrate by holding down the left mouse button and dragging the cursor across the area of interest and letting go of the mouse button. When you are finished click the save and return icon ((floppy disk with arrow).

Peak Picking:

To pick the peak locations click the **Pick Peaks** icon from the Process menu bar. This will open a peak picking menu bar. Using the left mouse button draw a box over the peaks that you want to pick being sure that the bottom of the box stays above the baseline. The bottom of the box will be the minimum intensity of the picked peaks and the top of the box will be the maximum intensity (only peaks the fit within the box will be picked). Click the save and return icon when you are finished with this process.

Plotting the spectrum

Once you are satisfied with your data it can be printed. This instrument uses a special graphics program for this purpose. The best way to start this program is to clicking the plot tab above the spectrum. This will start the program with your spectrum in the middle. There are four areas on the spectrum 1) spectrum, 2) parameters, 3) Bruker logo and 4) text. You can choose the portion of the spectrum that you want to work on first by putting the mouse in that area and pressing the right mouse button. A box will appear around the area with green squares on the border. After the first area is chosen with the right mouse button all subsequent areas should be chosen with the left mouse button.

Manipulating the spectrum

When you highlight a specific area a menu will appear on the left side of the screen. You can use the information in this box to manipulate your data. Each area will have a different set of things that you can do to manipulate what is on the printout. In order the print the spectrum when you are finished you must click on the small blue triangle at the top right of the box and select the print menu.

When you are finished be sure to replace the standard sample in the magnet.

Backing up your data

There are currently two ways to save your data. You can save your data to your Prism Account from the NMR's directory or you can create a OneDrive in your email account.

Saving data to your prism account

Anytime that you wish to transfer your data to your prism account you must be in a terminal window. This can be opened on the Bruker instruments by clicking the right mouse button in the window. A menu will appear. Click **Konsole**. A konsole window will open. You now need to be in the directory that contains your data. To get there type: For AVIII-400 (c3po) **cd /home/your user name/nmr .**

For AVIIIHD-500 (chewie) **cd /opt/topspin3.5pl7/data/your user name/nmr**
For all other Bruker instrument **cd /opt/topspin3.5pl5/data/your user name/nmr**
You can check if your data is there by typing ***ls*** which will list the files in the current directory.
To transfer the data to your prism account type the command below:

scp -r < the filename><your gatech email address>@scp.prism.gatech.edu:

The ***filenam***e is the name of the data file that is in your directory. Be sure to type in the name exactly as it appears in your directory. A short cut is to highlight the name and then click the middle mouse button. This will paste the name in the next line.

The ***email address*** is your personal Georgia Tech email address on the Georgia Tech prism computer. The ***: (colon)*** at the end of the line is very important so do not forget to put it in.

You will then be asked to enter your password. This is your **Georgia Tech email password** and not the NMR password.

While the data is being transferred you will see some information going across the screen indicating the transfer. If you do not see these lines it might mean that you forgot to put the **:** (colon) after the line.

Setting up and using your OneDrive account.

Open the Firefox browser on the desktop. Open up your email account using mail.gatech.edu. In the upper left corner there is a waffle (a square of 9 dots),.click on this and select OneDrive. When this opens, you can create a folder for your nmr data. This is where you should put all of your data.

On the top row of this window click on upload. You will now need to browse to the directory where your data is located ie. /opt/topspin3.5pl7/data/your nmr user login name/nmr.

In the upper left corner, you will see your home directory. To the left of the word home is the icon for the browse button which will take you to the main directory of the nmr computer. Starting with /home (Bruker 400, c3po) or /opt (all other Bruker instruments) go to the directory where your data is located and then click upload. Your file will appear in your nmr data directory.