DCIF NMR Training Guide
400 MHz
Bruker AVANCE III HD b400

last edit 3/9/2015

The Bruker AVANCE III HD 400 has a broadband (BBO) probe with a ^{1}H Channel and X-Channel tunable from (30-300 MHz). Both channels may be tuned automatically by the TopSpin 3.1 software. The 400 has variable temperature capabilities.

Conventions used in this guide are as follows:

- **Boldface** type indicates commands that are typed into the TOPSPIN command line or in a terminal window

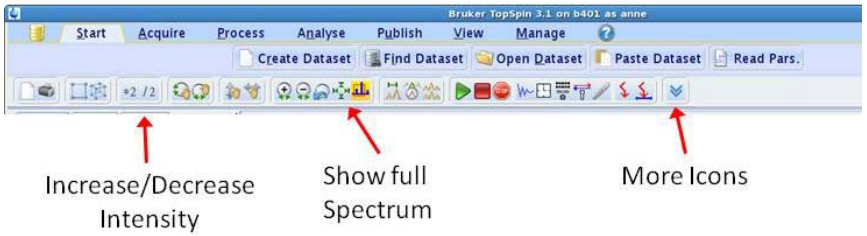
  Unless otherwise noted, all commands typed into the command line are followed by an <↵ Enter> keystroke.
- **LMB** indicates the Left Mouse Button
- **MMB** indicates the Middle Mouse Button
- **RMB** indicates the Right Mouse Button
- Table format: the left column contains the standard command line syntax, the right column shows button options.

Login and start the TOPSPIN software:

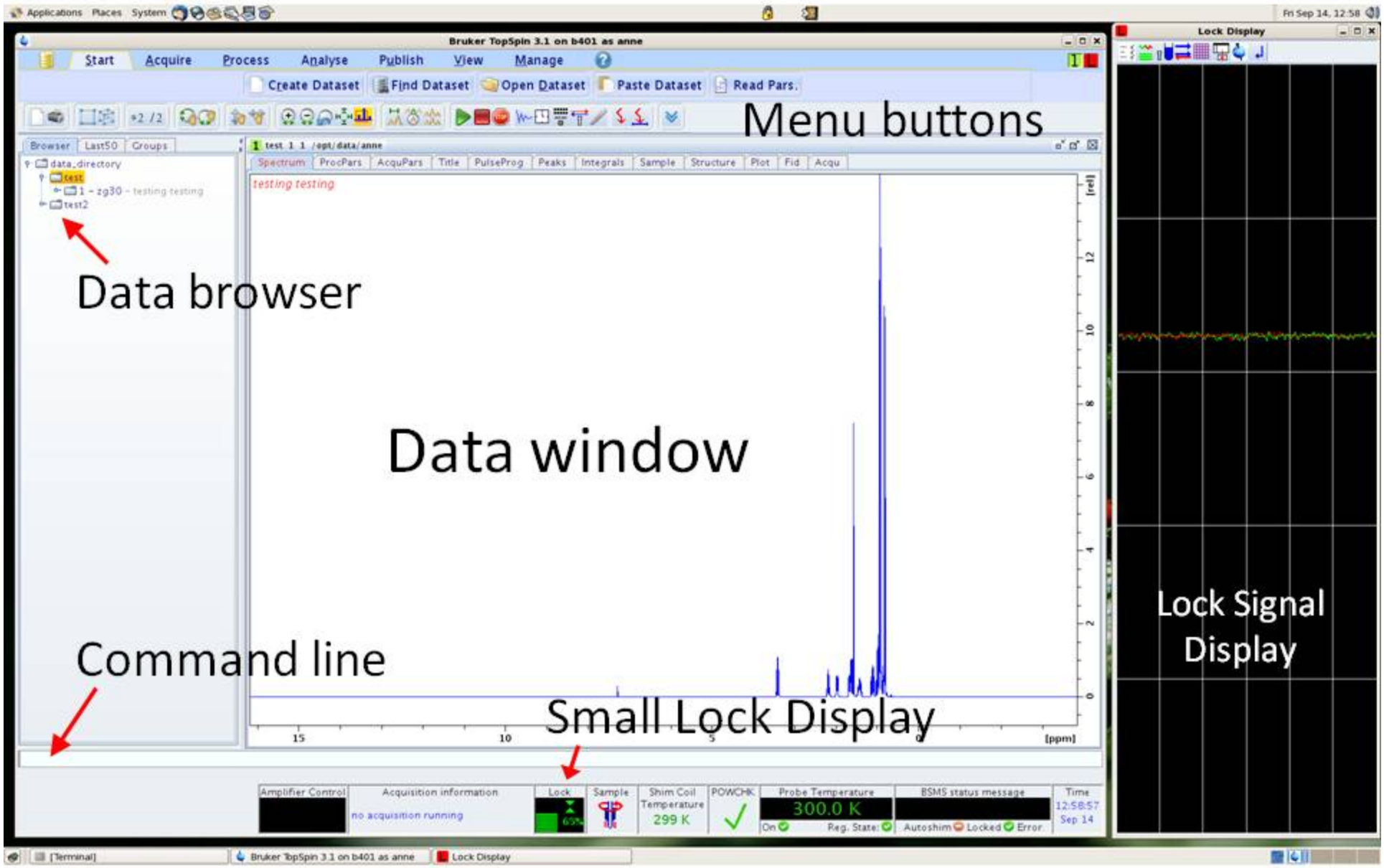
1. Login to b400, the HP host computer. If you do not have an account, access is not permitted. **No exceptions.**

2. Double click the TOPSPIN3.1 icon with the **LMB**.

3. Topspin 3.1 will open. Open the Lock Signal Display by right clicking on the Small Lock Display and selecting *Lock Signal Display*.

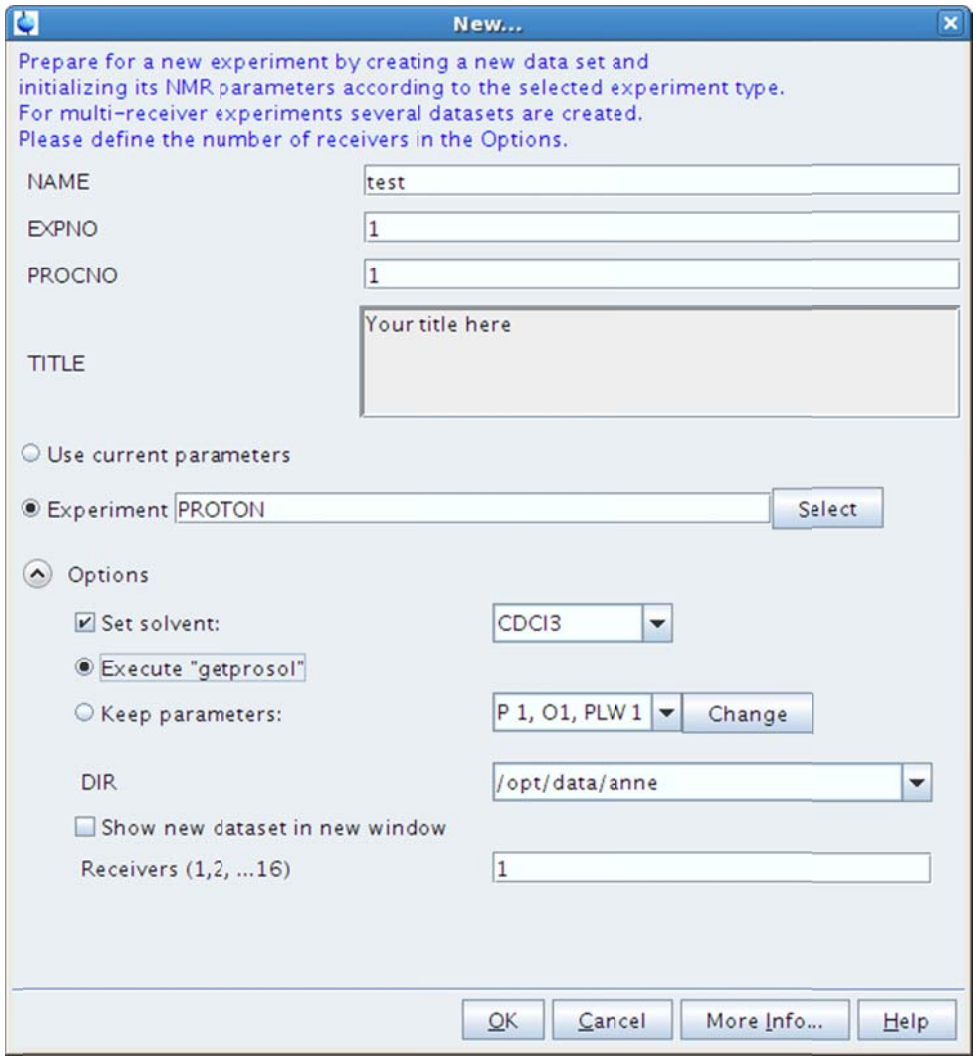


Some important menu buttons to remember! Hover your mouse pointer over a button, without pressing, and the software will tell you what the button does.


Menu buttons
Data browser
Data window
Command line
Small Lock Display
Lock Signal Display

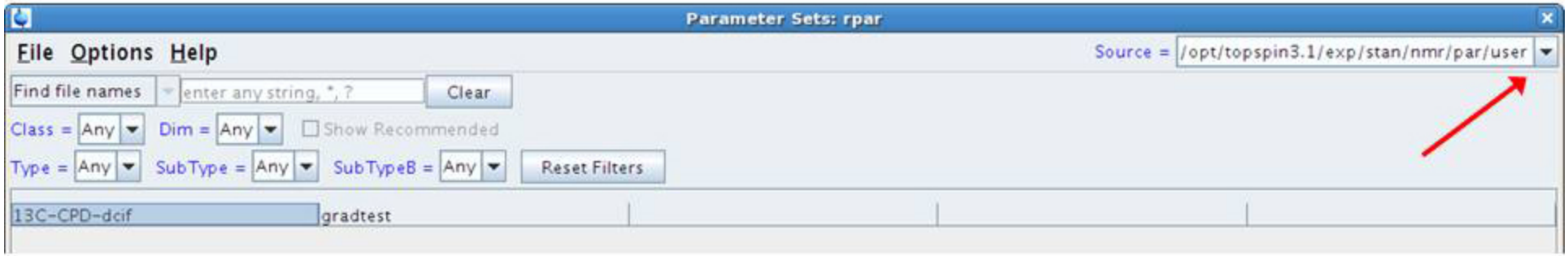
Setting up a new experiment:

1. Click (LMB) the Start tab in the upper left corner, then the Create Dataset button. You can also type **edc** on the command line. The New Experiment window will open. Fill in the desired name of your file, the experiment number (EXPNO), the processing number (PROCNO), and a title of your choice.



Make sure you click the Option arrow, check and set your solvent, and click Execute “getprosol”. The DIR should be /opt/data/<USER ID>.

2. Click the Select button to pick the experiment parameters you want to load. In the upper right corner of the *Parameter Sets* window, click the arrow next to Source, and make sure /opt/topspin3.1/exp/stan/nmr/par/user is selected. This will open the list of pre-saved *dcif* parameter sets. Select the one you want, then click *Set selected item in editor*.



The /opt/topspin3.1/exp/stan/nmr/par list is the list of default TopSpin parameter sets. These are unedited and straight from Bruker. You are welcome to use any of these if you would like.

Click OK when finished.

Inserting your sample:

1. Click the Acquire tab, then the Sample button. A drop down menu will appear. Select *Turn on sample lift air (ej)* or type **ej** on the command line.
2. Make sure you hear the air before you insert your sample in the magnet.
3. Click on the Sample button. A drop down menu will appear. Select *Turn off sample lift air (ij)* or type **ij** on the command line.

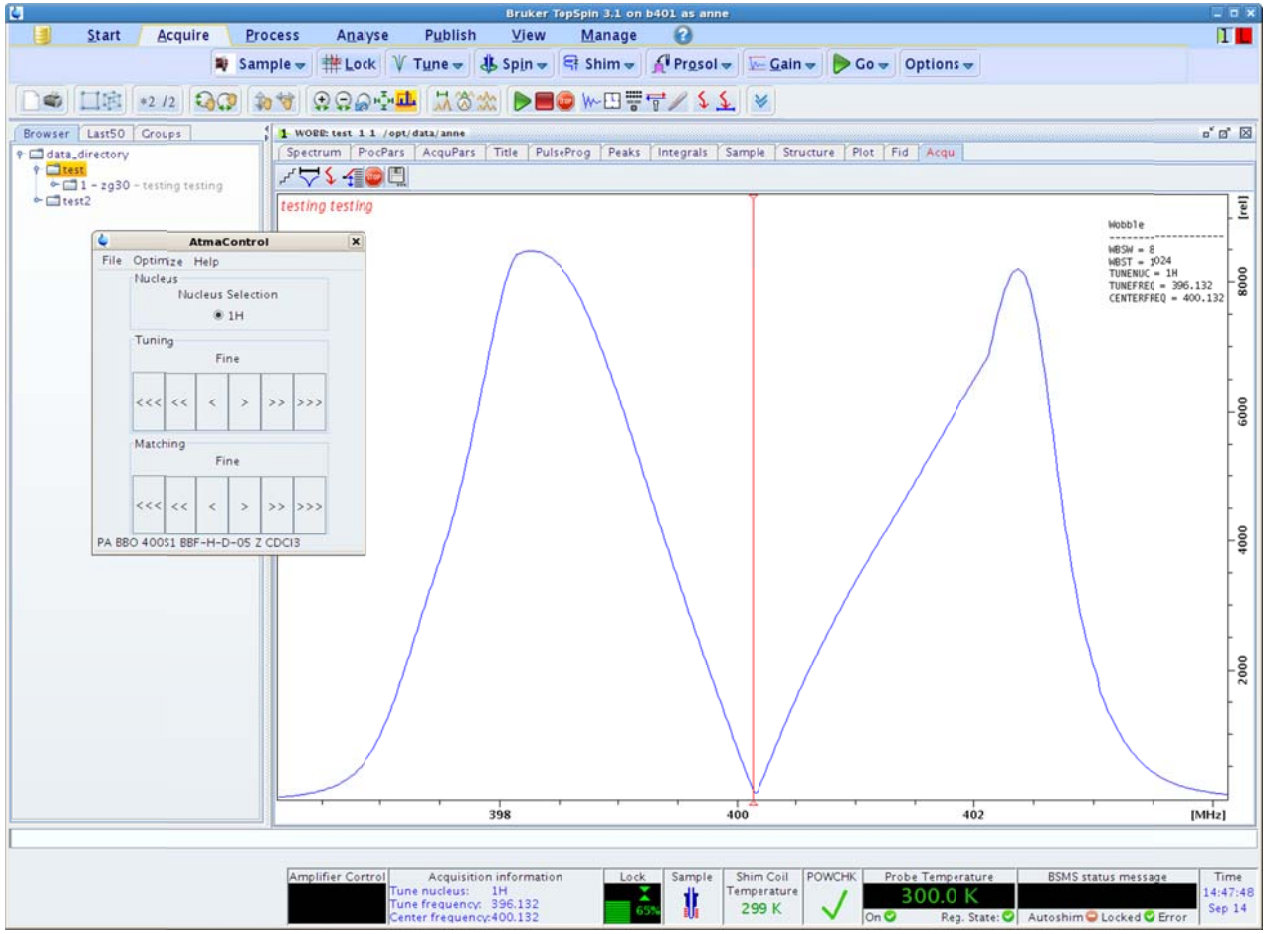
Locking:

1. Make sure the Lock Signal Display is open. If not, open the Lock Signal Display by right clicking on the Small Lock Display and selecting *Lock Signal Display*. You can also type **lockdisp** on the command line.
2. Click on the Lock button. Select your solvent from the Solvents table and click Ok. You can also type lock on the command line to open the Solvents table.
3. Wait for the lockn:finished message to be displayed under the command line.
4. **If you want to lock manually**, you can open the BSMS table by right clicking (RMB) on the small lock display, and then selecting the Lock/Level tab. You can also open the BSMS panel by typing **bsmsdisp** on the command line. Under the LOCK section, click the Field button. Using the Step

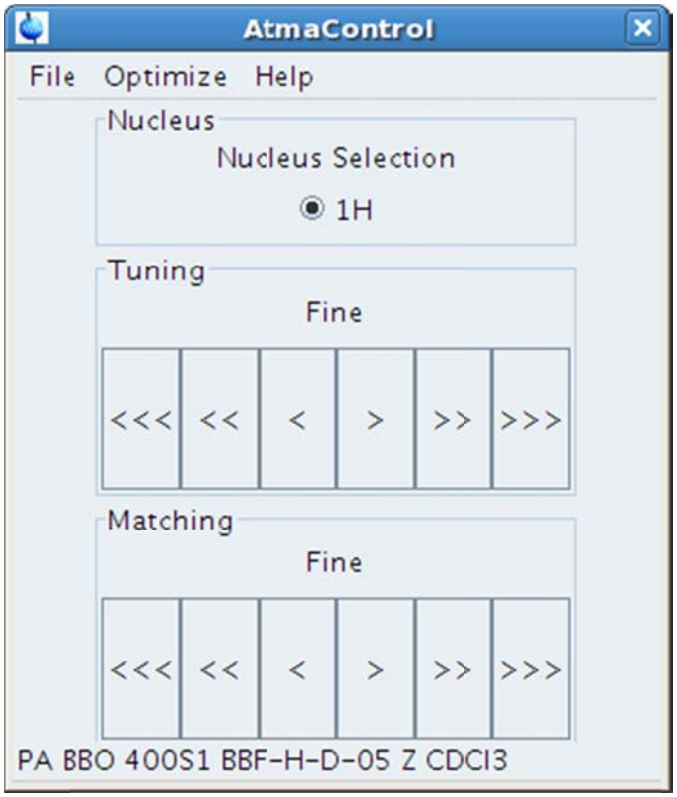
+ and Step – buttons, center the signal in the lock display, and then press the On-Off button. **NOTE:** if your solvent has multiple deuterium signals (CD_3OD, for example), you may want to lock manually.

Tuning:

1. Click on the Tune button to launch the auto tuning feature. You can also type **atma** on the command line. If you get an error message, type **atma** again.
2. BE PATIENT. Wait for the atma done message to appear below the command line.
3. If you want to tune manually, click the arrow on the Tune button, and a drop down menu will open. Select Tune/match ATM probe manually (atmm). You can also type **atmm** on the command line. The wobb and the Atma control will open.



4. If tuning manually, use the arrows on the AtmaControl to adjust the Tuning and Matching as needed. Remember you want the point of the wobb's "V" to be on the bottom horizontal line and centered on the red vertical line.



5. When you are done, click File and then Exit. If you are tuning for an unusual nuclei for the first time, you may want to Save Position before you Exit.

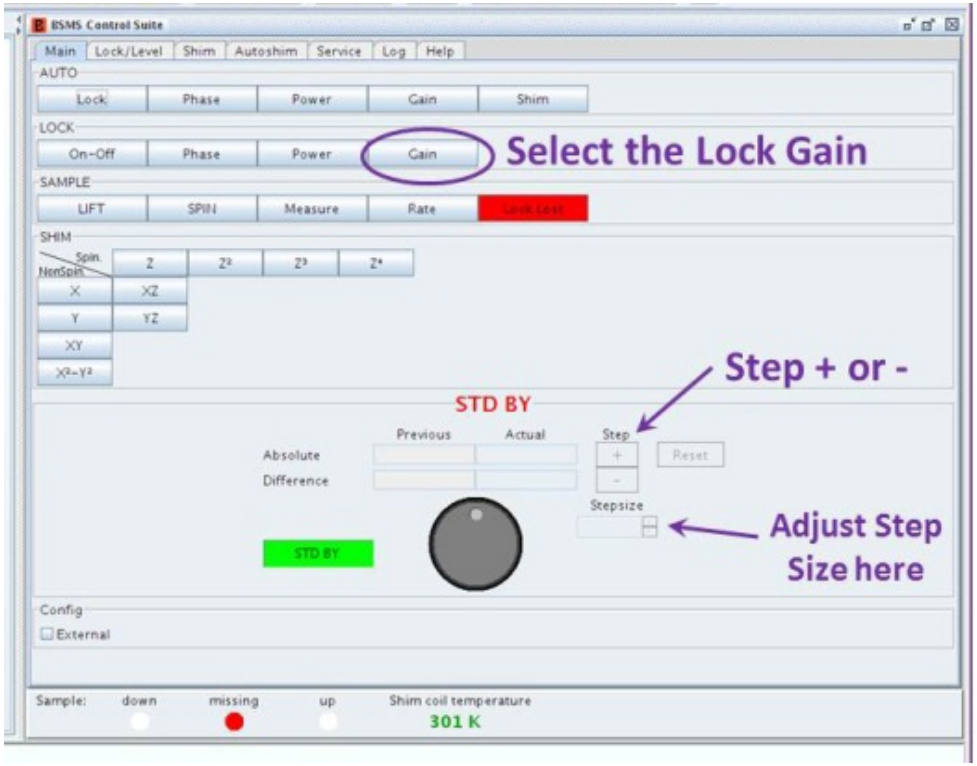
PLEASE NOTE: Every time you load parameters for a new nuclei, you MUST run **atma**. Simply loading parameters does not tune the probe. **atma** is an auto-tuning program, but the command must be entered for it to actually tune the probe to the desired nuclei.

Spinning:

1. Click on the spin button. A drop down menu will appear. *Turn sample rotation on (ro on)*. You can also type **ro on** on the command line.
2. If you want to use the BSMS table, just click the spin button in the Main tab. It will turn green when it hits the target spin rate.

Shimming:

1. Click the Shim button or type **topshim** on the command line.
2. **BE PATIENT**. This may take several minutes to finish. Wait for the topshim: completed message to be displayed under the command line and for your lock level to return to approximately the same level is was at before TopShim started (watch the Lock Signal Display). TopShim temporarily turns off the lock while it is running and will relock once it is finished.
3. If your lock level exceeds 100% (the lock level is at the top of the lock display window) after **topshim** has finished, you should open the BSMS panel (right click on the small lock display), and reduce the lock gain with the **Step -** button. Your lock level should be below 100% and above 50%.



4. You can still shim manually using the BSMS panel, if you wish. Click the Shim tab, and select which shims you wish to adjust.

Prosol:

1. If you did NOT check the Execute “getprosol” button when you created your dataset, click the Prosol button.

Setting the gain:

1. Click the Gain button or type **rga** on the command line.

Start the Acquisition:

1. Edit parameters as needed by either going to the AcqPars tab or by using the command line. For example, if you only want to run 8 scans, you can change that value for NS in the AcqPars tab to 8 (remember to hit Enter after you change a value) or by typing **ns 8** on the command line. Remember that **ns** must be a multiple of 8.
2. Type **ii** to initialize the system. Wait for this command to finish (watch for the ii: finished message below the command line).
3. Click the Go button or type **zg** on the command line. Wait for your experiment to finish.
4. You can process your data while the acquisition continues by typing **tr.** This will transfer the currently completed transients (scans) to disk.
5. Typing **halt** to stop the experiment at the end of the current scan. The data set will be saved. Typing **stop** will stop the acquisition immediately and your data will NOT be saved.

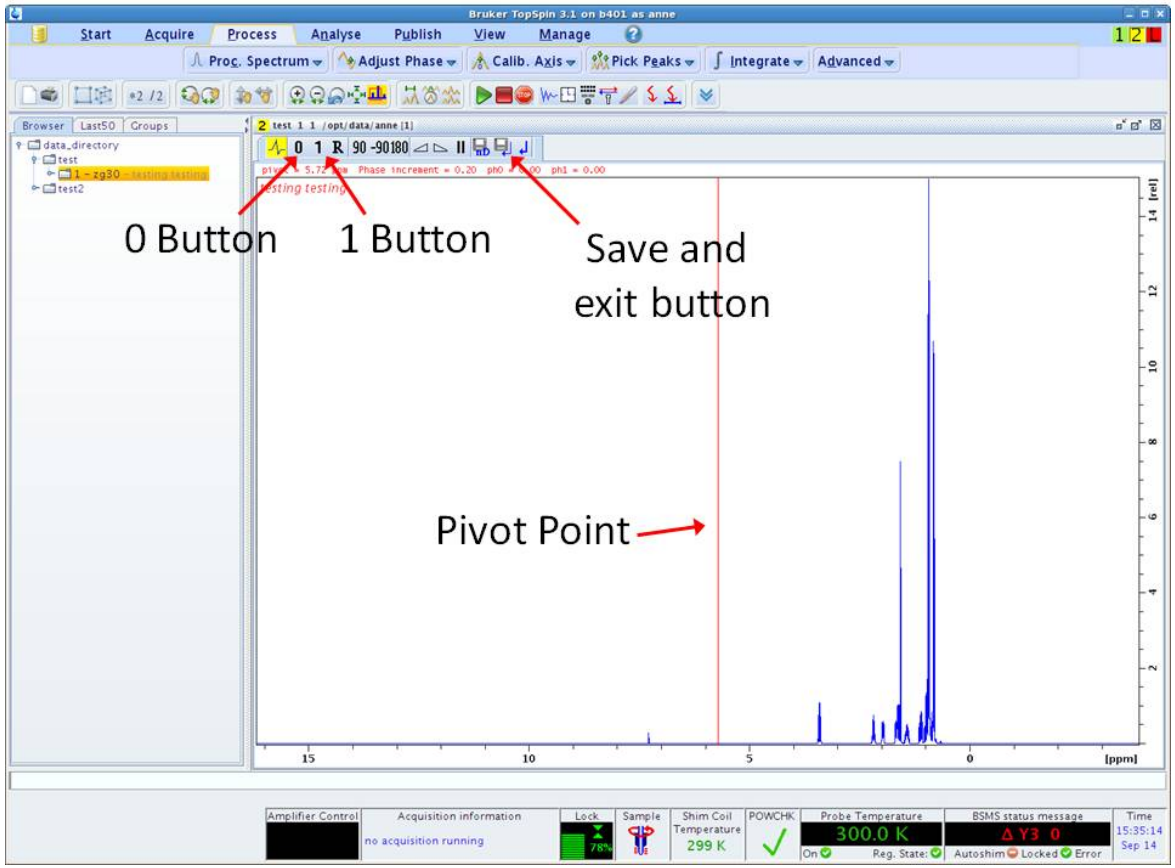
Processing your data:

1. It is recommended that you process your data in the computer room, using the workstations and NOT the spectrometer computer. Note that 400 data is stored in **/opt/data/<USER ID>.**
2. Click on the Process button, then Proc. Spectrum button. This will Fourier Transform your FID into a spectrum (ft), auto phase (apk), and correct the baseline (abs). You can also type **efp** or **ft** on the command line.

Phasing:

1. Click on Adjust Phase to enter the manual phasing mode.
2. The software will place the pivot point (red line) on the largest peak for you. If you wish to move this, click the **RMB** wherever you want to *set the pivot point.*
3. Hold the **LMB** down on the ***[0]*** button and move the mouse up and down. This will adjust the zero order phasing in the vicinity of the red vertical red line. Correct the phasing on peak closest to the red line and ignore the phasing elsewhere.

4. Next, hold the **LMB** down on ***[1]*** and adjust the phasing of the peaks farthest away from the red line. This is the first order phase correction. Think of the red line as the pivot in a seesaw or the fulcrum of a lever. The closer you are to the pivot point, the less adjustment you will see to the phasing. The farther you are from the pivot, the more adjustment you will see. You can move the pivot point as needed to help adjust the first order phasing.
5. It may take several iterations of adjusting ***[1]*** and ***[0]*** to get your spectrum phased. Click [***R***] to reset the phase back to its original.
6. To exit manual phasing mode, click the *Return & save phased spectrum* button (resembles a computer disk with a blue Enter arrow beneath it).

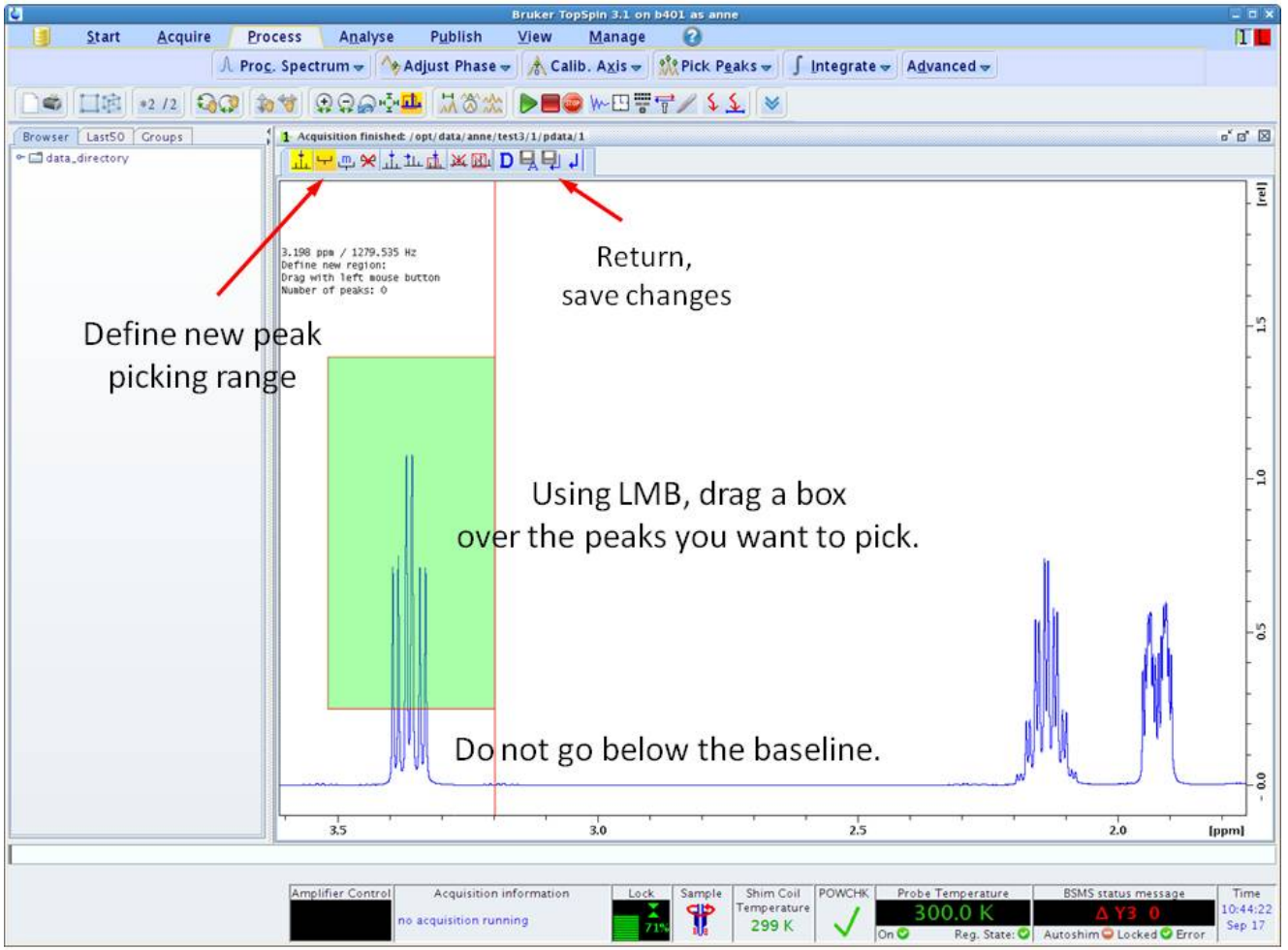


Calibrating your spectrum:

1. Zoom in on the peak you wish to calibrate (Left click, drag, then let go). Click on the Calib. Axis button. Select the solvent or reference pick by left clicking on it and entering the desired value. Click Ok.

Peak Picking:

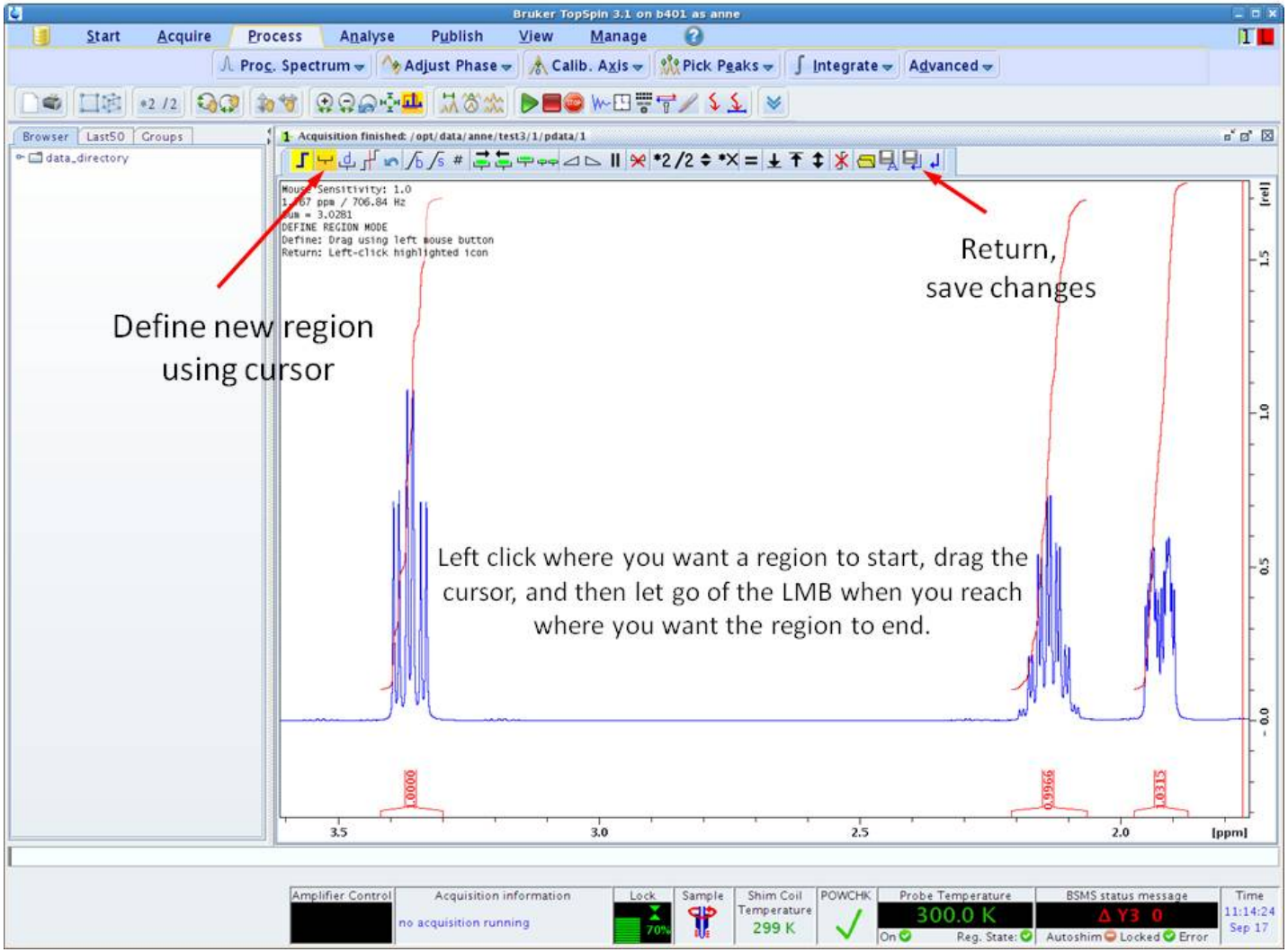
1. Click the Pick Peaks button.



2. Click the *Define new peak picking range* button (if not already selected). Using the **LMB**, drag the green box over the peaks you want to pick. Do not go below the baseline, or it may peak pick noise. You can select multiple regions by making as many green boxes as you want.
3. When you are finished, click the *Return, save changes* button to exit the peak picking mode.

Integration:

1. Click the Integrate button.



2. Click the *Define new region using cursor* button (if not already selected).
3. Using the **LMB**, click where you want an integral region to begin. Drag the cursor to where you would like the region to end, and let go of the **LMB**.
4. If you would like to delete an integral region, right click in the region and select Delete Current Integral.

5. If you would like to set an integral region to a specific value, right click in the region and select Calibrate Current Integral. Type in the desired value and hit Ok. Remember you cannot have negative integral values.
6. When you are finished, click the *Return, save changes* button to exit the integration mode.

Printing:

1. Click the Publish button.
2. Clicking the Print button will print the active screen (the screen as it is currently displayed).
3. The Plot Layout button will print the currently selected layout. If you click the arrow on the Plot Layout button, you can edit and modify the layout in the TOPSPIN Plot Editor.

Removing your sample:

1. On the command line, type **lock off** to turn off the lock, **ro off** to turn off the spin, and then **ej** to eject your sample. Type **ij** to turn off the air once you have retrieved your sample.
2. If you do not want to use the command line, open the BSMS Panel by left clicking on the Small Lock Display. Click the green LOCK On-Off button and the green SAMPLE Spin button. Then click Lift to eject the sample. Remember to click Lift again to turn off the air, once you have retrieved your sample.
3. Type exit on the command line to close TopSpin.
4. To log off the spectrometer computer, click on System in the upper left corner and log yourself out.

Other options:

1. Feel free to explore the other features offered in the Analyse and View tabs. If you are curious about a button, hover your mouse pointer over a button, without pressing, and the software will tell you what the button does.