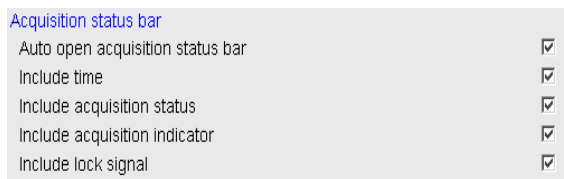


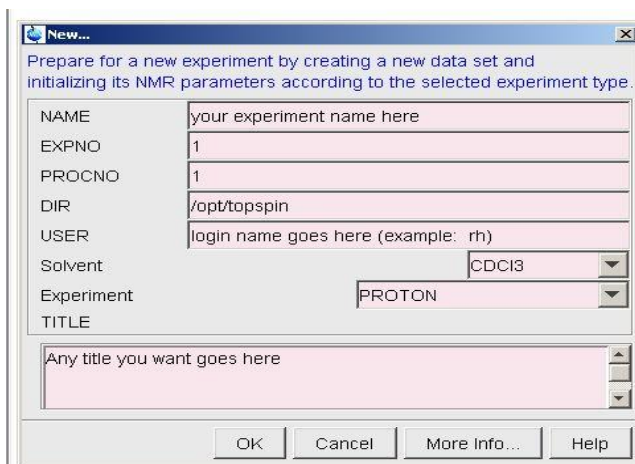
# Quick Guide to Topspin

1. Log into the workstation. Right mouse click on the desktop anywhere and select 'new terminal'. Type **topspin** [enter] (if this fails, create the launcher as described next). Alternatively, you can right mouse click on the desktop and select 'new launcher' or 'new link to application' depending on the operating system version. An interactive box will launch, type NMR in the top box, go to the 'command' box and type **/opt/topspin/topspin** and click the little box that says 'run in terminal', and then select 'ok'. If you are successful, there will be an icon on your desktop named **NMR** that you can click each time you log in to launch Topspin.

2. The first time it will ask you if you agree to the license agreement. You must select 'yes' to proceed. Type 'setres' [enter] in the command window at the bottom of the Topspin screen. A new box will pop up. Check the box at the very top of the screen. Also, if you would like to see the 'acquisition status' of your experiment, scroll down the preferences screen and check all the boxes in the acquisition status section that are shown in the figure to the right. Exit Topspin by typing 'exit' [enter] in the command box. Re-enter.

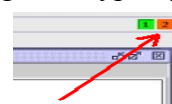


3. Type 'edc' [enter] in the command box at the bottom of the screen. A new window will pop up asking you for a filename, experiment number (EXPNO), process number (PROCNO), directory for data storage (DIR), username (USER), solvent (select your solvent from the list), and Experiment (select from list). Typical 1H and 13C experiments are PROTON and C13CPD, respectively. If you pick the experiment in this section you do not need to 'rpar' one later. You may add a title to your experiment in this section. Click 'ok' when you are finished.



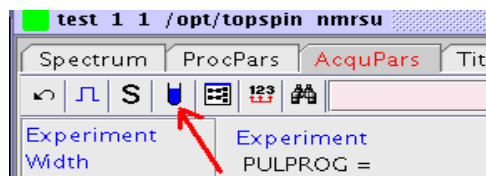
4. You can rpar whatever experiment you like now if you did not select one in the 'edc' window.

5. Place your NMR tube in the spinner and set the appropriate depth with the gauge located at each spectrometer. Type 'ej' [enter] and exchange your sample with the one in the magnet. Type 'ij' [enter] to lower your sample into the magnet. Type 'lockdisp' [enter] to launch the lock display. The lock display window automatically minimizes itself after launching. In order to see it, click the little orange box with the number 2 in it up in the right hand quadrant of the window. Type 'lock' [enter] in the command window and select your solvent. Once locked (the green light should be solid under the lock button on the bsms panel beside the workstation), you will need to shim on the sample by adjusting the z and z2 shims. Push the 'z' button on the bsms panel and turn the knob in either direction while monitoring the lock signal. Improvements in shimming are evidenced by the horizontal lock signal line moving upward toward the top of the screen. When the horizontal line reaches the top of the screen you need to press the lock gain button and reduce the value until the line is back near the 70% level of the screen size. Continue to maximize the signal moving back and forth between **z** and **z2**. Adjust **lock gain** or **lock power** (push buttons) to keep the horizontal line in the display window. This whole process should take no more than 3-5 minutes. If after you have shimmed, you still cannot attain a good lineshape (expand around the solvent peak in your spectrum to check the lineshape), you can recall a recent shim file by typing **rsh** [enter] and select the shim file your solvent that has the suffix

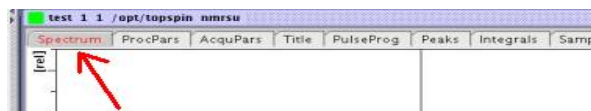


most recent (if you do not see your solvent, start with acetone\_most\_recent....you will still need to touch up z1 and z2 after retrieving the shim file. Notice the shimming guide taped to the table next to all spectrometer workstations for which shim to adjust for certain artifacts.

6. You can either type 'eda' [enter], or just click on the little tab that says AcquPars. This is where you will adjust the number of scans. You should have already specified the solvent in the 'edc' window. But if you want to double check, scroll to the bottom of the AcquPars window and you will see the solvent selection option. You will notice a little blue button that looks like a test tube at the top left of the eda window. Left mouse click on this, it is the getprosol button. Alternatively, you could just type 'getprosol' in the command window at the bottom of the screen. Click the 'Spectrum' tab when you are finished.

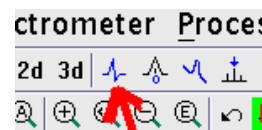


7. Type 'rga' [enter]. Wait ~30 seconds. When it says it is finished you can run your experiment by typing 'zg' or 'zz' [enter]. If you type zg, you will need to type 'efp' [enter] when the acquisition is finished to see your spectrum. If you are running a longer experiment and want to view the spectrum before the acquisition is finished, type 'tr' [enter], and then wait ~10 seconds and type 'efp' [enter].

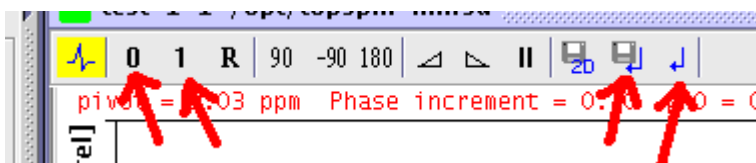


**NOTE!!** If you are doing 13C, don't forget to do the 'atma' procedure on Zeus and Hades, or 'wobb' for Artemis and Ares. This procedure can be found on the last page of these instructions.

9. You can phase your spectrum with 'apk' [enter] or manually. To manually phase the spectrum, click on the icon shown to the right which will launch an interactive phase window. You will see a 0 and 1 at the top



of the screen. Hold the left mouse button down and scroll up or down on either of these two buttons to phase manually. You will see a little arrow and a disc on the far right hand side. Click this to save your phase correction and return. If you click the little arrow that does not have the disc, your correction will not be saved.



10. To integrate, click on 'Analysis' and select 'integration'. An integration manager will launch giving you options to integrate manually, or automatically, etc. If you select manual, scroll the mouse over the buttons at the top of the integration manager screen to determine what they do (it is fairly intuitive). The second icon from the left allows you to manually use the cursor to define the integration area. Right mouse clicking on any integral gives you the option to calibrate or delete it. When you are finished, click the little arrow with the disc like you did for the phasing (step 9 in case you used apk) to save your integration.
11. Peak picking is done in a similar fashion, select 'Analysis' and 'peak picking' and you can choose to do it manually or automatically, etc. You can change the threshold options for the min/max intensity, sensitivity, limits, etc. with the automatic option. If you choose manual, you will need to scroll the mouse over the buttons to determine their function and save your work when you are finished.
12. To print, click on the little printer icon at the top left. You will see three options. The first is print the active window. This will give you a spectrum with title only in a 'what you see is what you get fashion', no parameters. The second option, 'print with layout' will launch xwinplot and you will

have to select 'print' again from there to print and the result will include any edits you have made using xwinplot (NOTE: numlock must be off to edit in xwinplot, right mouse click to select edit when numlock is off). The third option, 'print with layout-plot directly' will print your spectrum with peaks, integrals, and parameters, but it will automatically scale the spectrum so the tallest peak (even if it is the solvent peak) will be at the top of the spectrum. If you get an error here, you will need to type 'edp' and find the 'layout' specification box in the automation section, and select 1D\_H+pp.xwp.

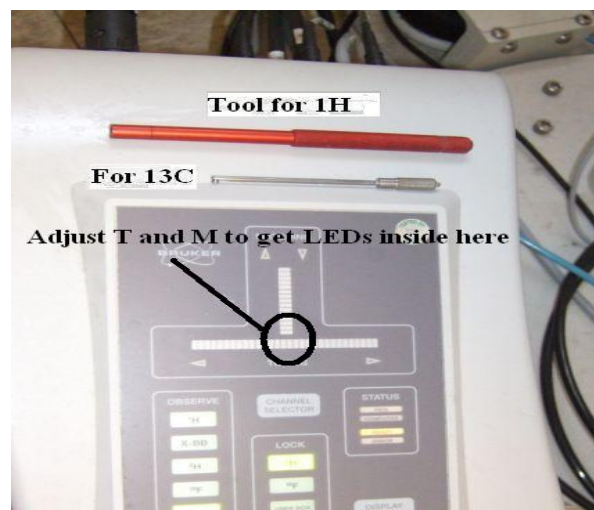
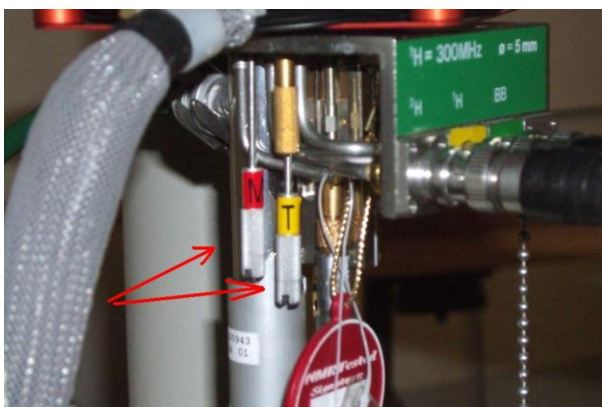
13. If you scroll the mouse over the various buttons on the screen, it will tell you what they do.
14. When you are finished, eject your sample with the 'ej' [enter] command and return the standard sample (chloroform in Acetone-d6) to the magnet. Type 'lock' and select 'acetone'. When it is finished, you can type 'exit' in the command line, or just hit the X in the top right hand corner of the main window. Do not forget to log out of the workstation.
15. To log off the workstation, left mouse click on the red hat icon (bottom left) and select 'log out'.
16. Please sign the logbook next to the workstation

PROCEDURE FOR TUNING AND MATCHING IS ON THE NEXT PAGE

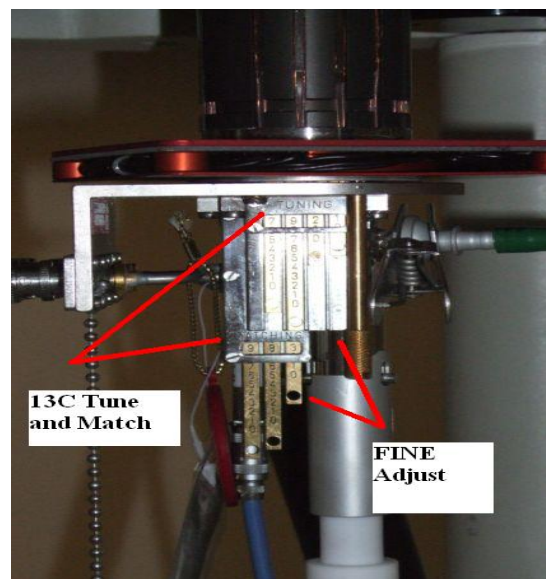
# Tuning and Matching on Artemis and Ares

The quality of the tuning and matching has a significant impact on the sensitivity of the  $^{13}\text{C}$  spectrum and 2-D experiments. It can also affect  $^1\text{H}$  sensitivity if it is way off.


1. **DO NOT PROCEED IF YOU HAVE NOT BEEN TRAINED TO DO THIS STEP!!** If you do not know how to optimize these settings and would like to learn, please contact Russ (X33069) and he will show you.
2. Be sure you are in the appropriate experiment number. Type rga [enter]. When rga is finished, type 'wobb' [enter] in the pink command window.
3. For  $^1\text{H}$ , use the small red hollow tipped tool on the HPPR to adjust the two screws labeled T and M for tuning and matching. Watch the little readout on the HPPR to assess your progress. Ideally, you want all green lights inside the circle in the diagram below.

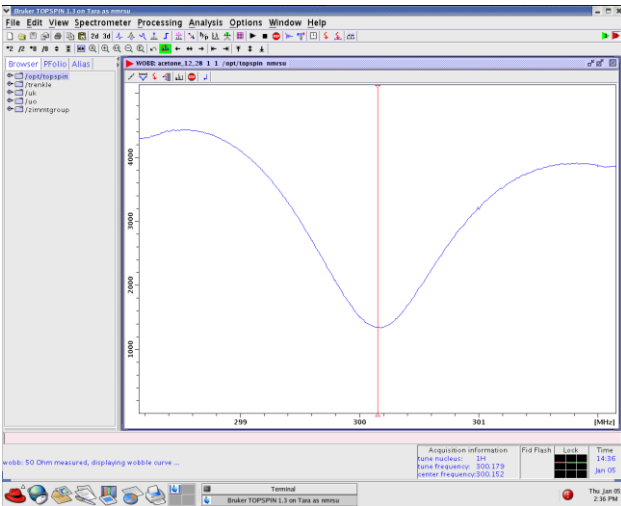


4. For  $^{13}\text{C}$  or other heteronuclei (Ares only), adjust the sliders labeled Tuning and Matching under the probe with the small silver tool on the HPPR trying to attain the same goal as for  $^1\text{H}$ . For Artemis, it is the blue screws similar to the  $^1\text{H}$  screws for tuning and matching  $^{13}\text{C}$ . NOTE: There is a small card under the probes with values of tuning and matching for most nuclei of interest. This is a good place to start if the settings are way off (way off if you don't even see a dip in the 'wobb' window in the Topspin screen). If you do see the dip, then the FINE sliders should be all you need to adjust.

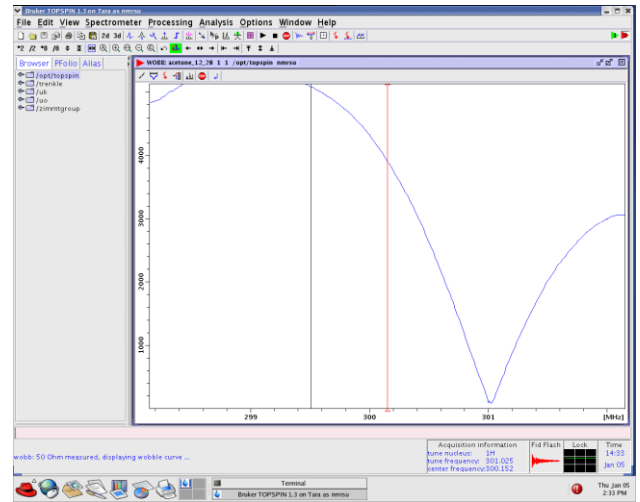


5. NOTE: When tuning and matching for any indirect detection experiment (HSQC, HMQC, or HMBC), you will need to check both nuclei,  $^1\text{H}$  and  $^{13}\text{C}$ . To switch between applicable nuclei,

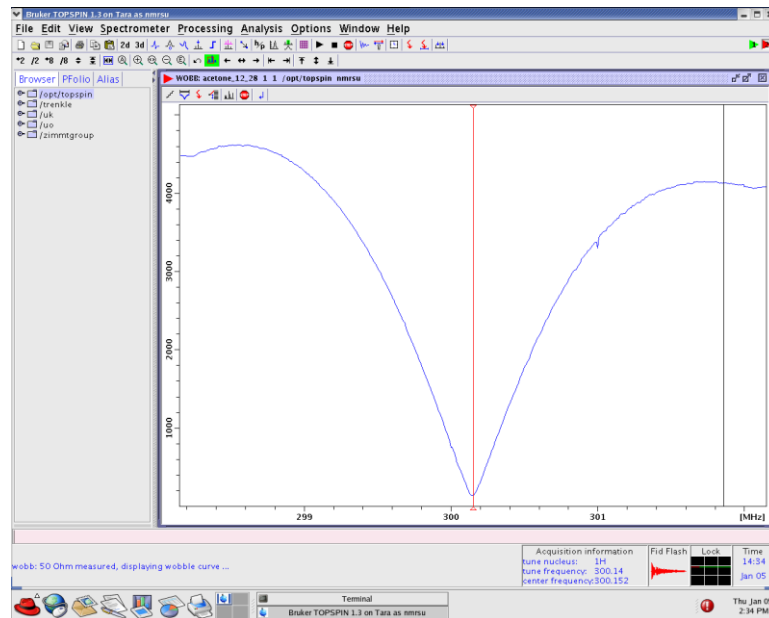
click the  icon. When finished, hit the return symbol in the wobb window.



Tuned, not Matched



Matched, not Tuned



Tuned and Matched

NOTE: IT MAY NOT BE POSSIBLE TO ATTAIN PERFECT TUNING AND MATCHING AT ALL TEMPERATURES. WHEN THIS OCCURS, IT IS BEST TO DETERMINE YOUR NEW 90° PULSE WIDTH EXPERIMENTALLY BEFORE ATTEMPTING TO RUN ANY EXPERIMENTS.