General Procedure for XWinNMR/TopSpin

DO NOT approach the super-conducting magnet with any credit cards, analog watches or loose metal objects.

1. **LOG ON**. If another user is logged on, close <u>all</u> open programs and log out the user before logging on to your personal account.
2. **OPEN** the TopSpin software by double-clicking on the desktop link
3. **PLACE** your sample in a spinner and use the depth gauge to set the appropriate depth. The sample should be centered around the 5mm coil line. Put your sample in the spinner. Measure in the depth gauge. **Sample must not touch the bottom of the gauge.**
4. **INSERT SAMPLE**: Press the "LIFT ON/OFF" button on the BSMS keyboard. WAIT. It will be several seconds before the air comes on. When the standard sample appears at the top of the magnet bore, remove it and insert your sample. Turn the lift air off with the "lift on/off" button. Make sure that the spin button is lit. It will take about a minute or more for your sample to insert and begin spinning. Be sure to check that your sample is spinning.
5. **LOCK** your sample. Type "*lock*" on the command line and select your solvent in the window that opens. You can observe the graphical display of the lock by typing "*lockdisp*" on the command line, or by selecting "Lock" from the "Spectrometer" menu.
6. **SHIM.** Type "*gradshim*" on the command line. A window will open. Click "Start Gradient Shimming". This process takes 2-3 minutes. At the end of which, the lock signal will be higher than before. The lock level may be off the top scale of the display. In which case you should press the "LOCK GAIN" button on the BSMS keyboard and use the wheel to turn the gain down until it is about three-fourths of the scale. If results are not good, load a standard shim set, "*rsh current*", and redo the gradshim procedure.
7. **CREATE** a new experiment. Type "*edc*" or select "New" from the "File" menu. Enter a sample name, or the date if you prefer, set experiment number to one, and make sure that the data is stored in /opt/topspin/ and that the username is your username. Select your solvent and the parameters for the experiment that you would like to run from the pulldown menus. *Only* select a set of parameters that begins with "AAACRYO". These should appear at the top of the list. All other sequences are not ready to use without skilled set-up.
8. **EDIT PARAMETERS:** "*eda, ased, sw, ns, rg*", etc: if necessary, edit experimental parameters using the menus, or directly by typing the parameter name on the command line.
9. **ADJUST GAIN** "*rga*": automatically set receiver gain (for proton, unreliable for ^{13}C).
10. **TIME:** check your approximate experimental run time with "*expt*".
11. **START** the experiment "*zg*".
12. **ERROR?** If there is a problem these commands will end the experiment before completion: "*halt, stop, kill*". Use "*stop*" unless you know the difference between these commands.
13. **PROCESS** your FID to a frequency domain signal "*efp*", use "*xfb*" for 2D data.
14. **PHASE** correction: "*apk*".
15. **BASELINE** correction: "*abs*".
16. **ANALYSIS**: Calibration, integration and picking peaking: use the toolbar icons.
17. **PLOT**: "*view*" and "*plot*"/"*prnt*", or "*xwin-plot*". Remember to set the left and right limits on your plot if you have zoomed ("*dpl1*"). Edit plot parameters with "*edg*".
18. **EJECT** ("*ej*") your sample and REPLACE it with the standard sample. INSERT the standard sample and press the "*autolock*" button on the BSMS.
19. **CLOSE** all open programs including Topspin.
20. **LOG-OFF**. Close <u>all</u> programs and logout.