pKa Experiment
Chem 343 NMR Lab for Bruker DPX-400
UIC Chemistry RRC Building

Logging into the computer and starting the NMR software **TOPSPIN 1.3** on Linux CentOS

1. In the login window
   login:
   password:

BOLD lettering is typed on topspin command line or are keys pressed on board to right**.**

The BSMS Panel
Changing the samples and shimming using the BSMS
- have the TA help you with these steps
- it is critical not to drop a sample into the magnet without hearing the air as the sample will free fall down and break in the probe.

On the bsms board to right: a) **Spin-Off** b) **Lock-Off** c) **Lift-On.**

Sample should be swapped out, CLEANED with kim wipe, and set to probe depth using the sample gauge.

-make sure you hear the air is on before placing the sample into the magnet.

Insert sample: a) **Lift-Off** .

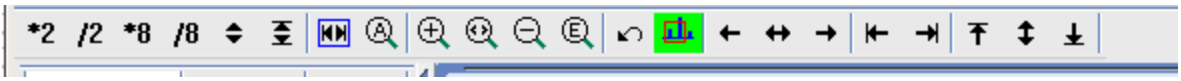
rsh shims.bbo This will read in standard shim files.
lock H2O and the spectrometer will lock onto the solvent (wait until finished).
lockdisp Launches lock display window.

Shim
Z1 and use wheel to maximize lock signal. Then **Z2** and do the same. Repeat 2 times.
STDBY key to put the bsmsboard (pic above) into standby mode.

Proton NMR data acquisition
edc - Add the name as you like eg) sample_1. This creates folder of your experiment.
rpar h1_es.bbo all – reads in 1D 1H experiment.

rga - sets the receiver gain automatically and takes several seconds to do so (wait..).
zg - starts experiment and overwrites current data). If pop up cl. **OK** to overwrite.

Processing:
efp (when experiment finishes we fourier transform and process the data).
apk (autophase the spectrum, all peaks should be pointing up now). Or try manual phasing :
Adjust scaling etc with:
setti -include appropriate title
pps – autopick peaks
export – save as *whatevername.jpg* and so on. Ok to create directory if you like.



or

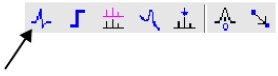
☞ Click one of the following buttons:

- *2 Increase the intensity by a factor of 2 [***2**].
- *8 Increase the intensity by a factor of 8 [***8**].
- /2 Decrease the intensity by a factor of 2 [**/2**].
- /8 Decrease the intensity by a factor of 8 [**/8**].
- Reset the intensity [**.vr**].

Manual Phasing if Needed (ie **apk** did not work well above)

How to Switch to Phase Correction Mode

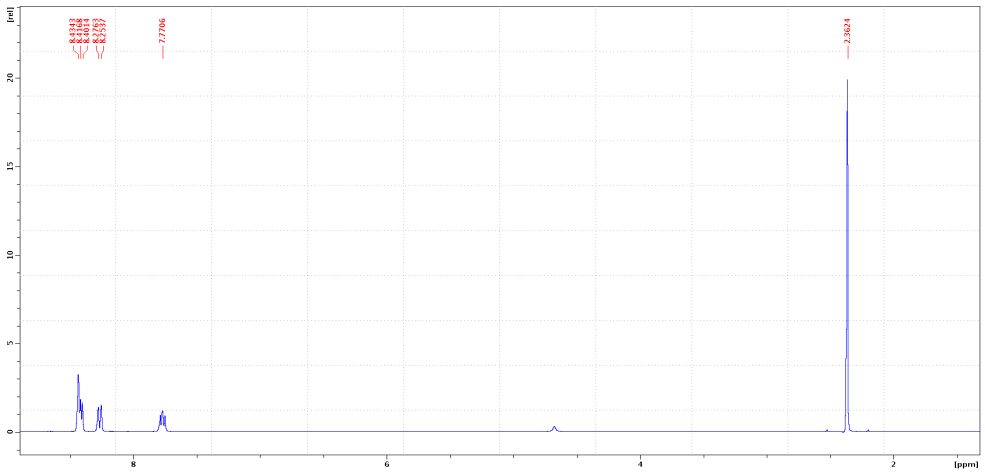
Click the indicated button in the upper toolbar:



Click-hold the button 0 and move the mouse until the until the reference peak is exactly in absorption mode.

Click the button to save and execute the phase correction and return.

Your resulting spectrum should look something like:



*We will acquire a

series of 1Ds like above and generate a 2D stacked plot when finished.

<u>Acquisition of additional experiments</u>
Eject sample and load new NMR tube:
Create 2nd exptl data set:

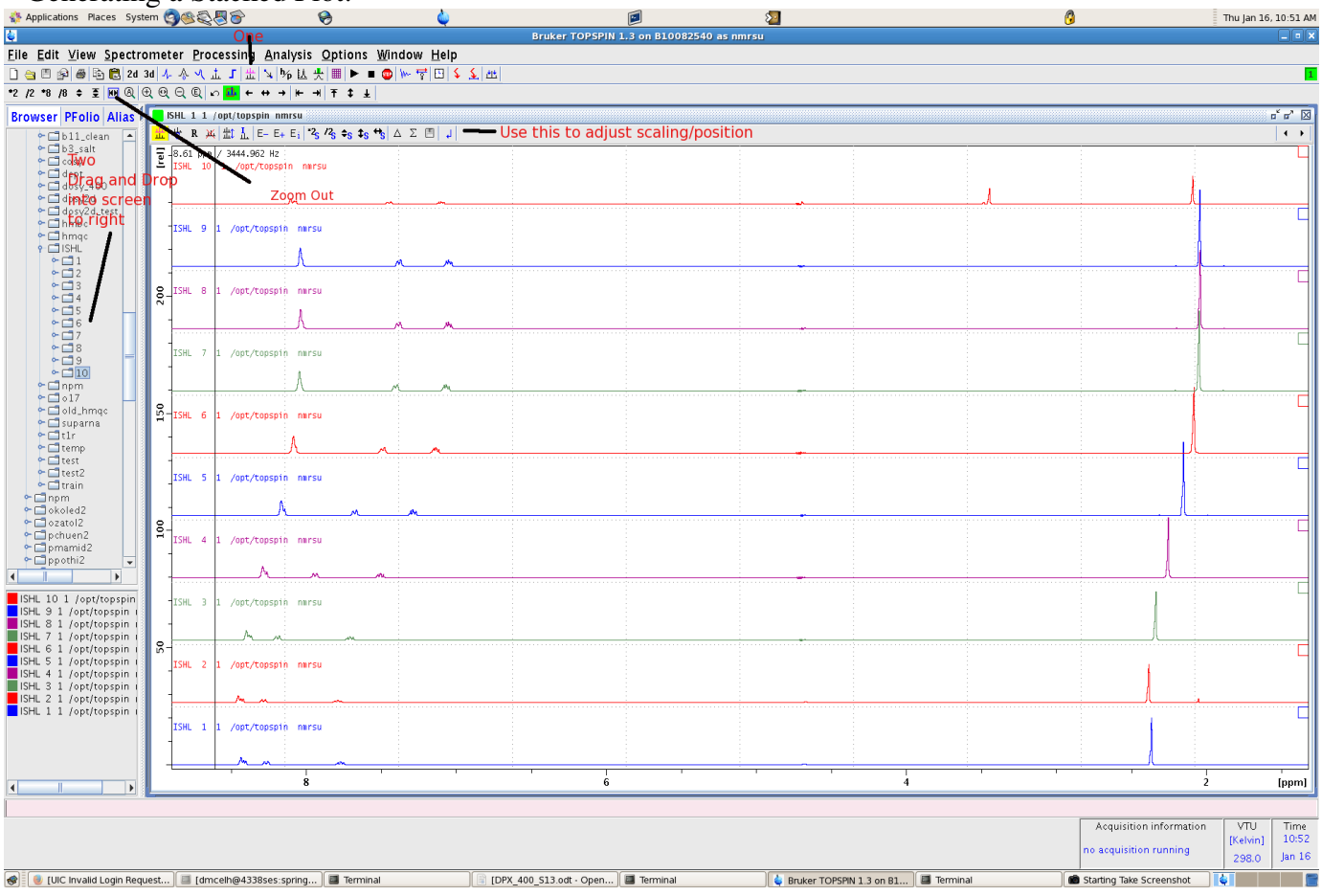
edc and edit EXPNO entry to 2.
rpar h1_es.bbo all – reads in 1D 1H experiment.
rga - sets the receiver gain automatically and takes several seconds to do so (wait..).
zg - starts experiment and overwrites current data). If pop up cl. **OK** to overwrite.

* you can also change quickly between these expts once created by: **re 1** or **re 2** etc. You are simply moving in the sub-directories of whatever the file was initially named.

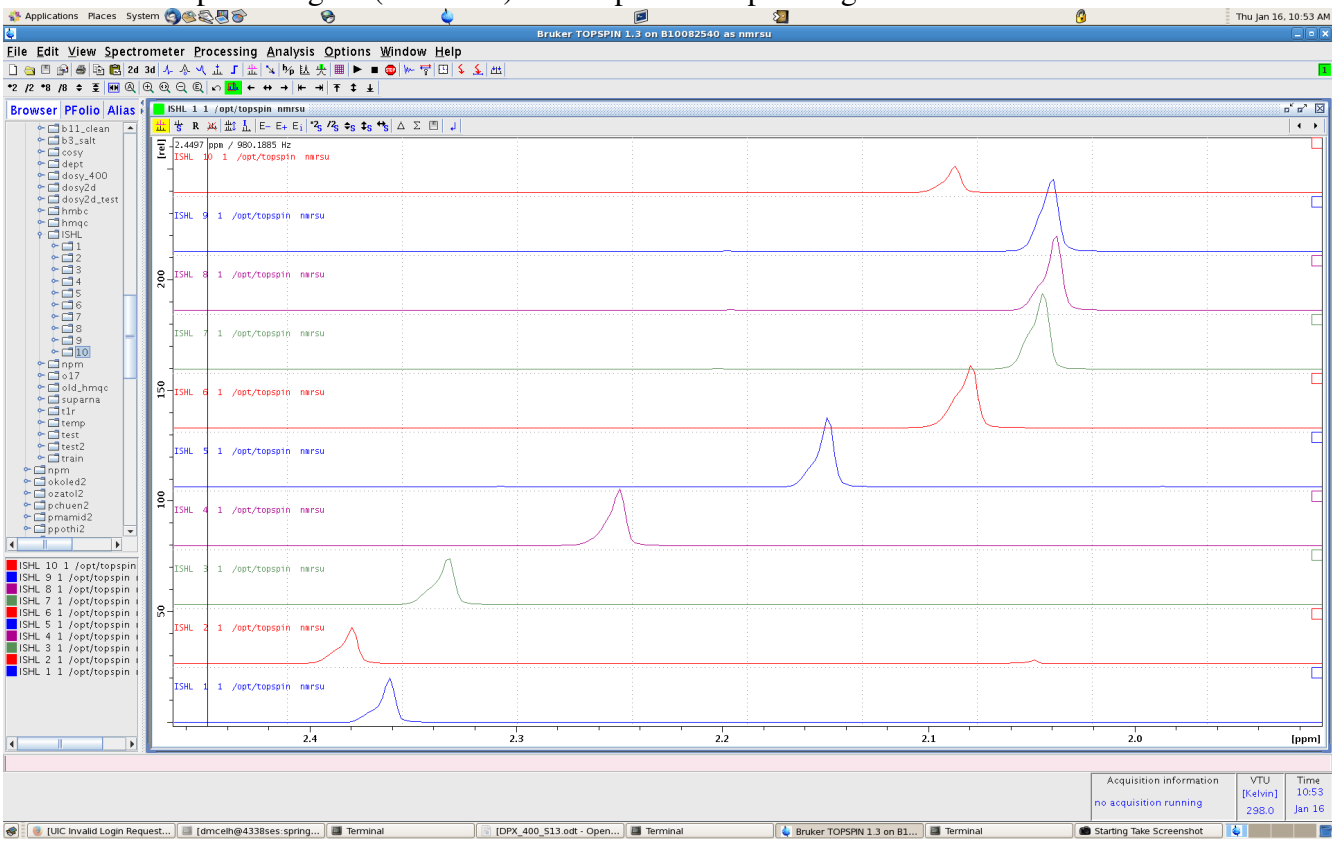
efp - process the data
apk - autophase. Now adjust peaks intensities so they fit to screen. use the *2 or /2 buttons.
Or phase manually again as well.
setti -include appropriate title
pps – autopick peaks
export – save as *whatevername.jpg* and so on.

*Now do the next sample *etc* until all have been acquired.

Generating a Stacked Plot:



-Use **One** in picture to select icon to start stacking mode
-Use **Two** and drag and drop the expts into the window on the right. Do so in order of data sets collected.
-We can left click and zoom into regions of interest and export .PNG files for your report.
-Zoom into Aliphatic region (low PPM) and export stack plot. Eg:



Note the expt 1 and 10 above look to be off, and probably shouldn't be included in the data fitting in this case.

-You can check the shifts right now by hovering the cursor over the peaks and record them into your note book if you like.

<u>When finished email yourself the data</u>

*data .jpgs/.png are stored on desktop 'chem343's Home' ..

<u>Finishing up with TOPSPIN and logging off the computer</u>

Remove your sample and replace it with the sample which was originally in machine
Type **exit** on command line to leave the NMR program.
logout icon (> type arrow) is on top bar.