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**.**

Additional information regarding topspin is on the Desktop in folder 'TOP_processing_a4.pdf' or 'Topspin1p3_users_guide.pdf'. Focus mainly on the processing, such as integration and phasing, if you get stuck.

Changing the samples and shimming
- 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 gauger.

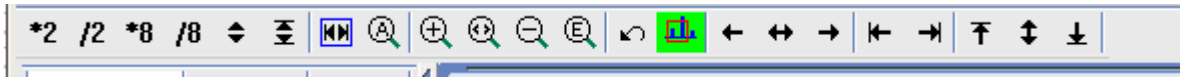
-make sure you hear the air is on before placing the sample into the magnet.

Insert sample: a) **Lift-Off** b) **Spin-On**.

rsh shims.bbo This will read in standard shim files.
lock and enter your solvent.
lockdisp Maximize lock (Shim) on bsms board below:

Shim
Z1 and use wheel to maximize lock signal. Then **Z2** and do the same. Repeat 2 times.
STDBY key to put the bsmsboard 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.bbo all – reads in 1D 1H experiment.
ii - this is used to initiate the interface or reset communications to the nmr.
rga - sets the receiver gain automatically and takes several seconds to do so.
zg - starts experiment and overwrites current data). In 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:



or

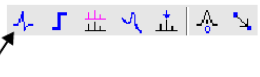
☞ Click one of the following buttons:

- *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**].

Phasing

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.

Integration/peak picking:
abs – baseline correction
int – auto integrate; use *auto-find regions*
in the integration mode (above), right click over integral region to calibrate the number of protons.
pps – autopick peaks
export – save as *whatevername.jpg* and so on. Ok to create directory if you like.

*Chapter 11.2 of 'Topspin1p3_users_guide.pdf ' has much more detail regarding processing if needed.

Acquisition of 13C NMR spectrum

Create 2nd exptl data set:
edc and edit EXPNO entry to 2.

Note it is common practice to use the experiment # entry as:
1: 1D 1H
2: 1D 13C
3: Dept135 and so on. just be sure to keep track in your notes.

* you can also change quickly between these expts once created by: **re 1** or **re 2** etc...

edc – create expt 2
rpar c13.bbo all

We use very similar steps as in the 1H expt.
ii
zg (experiment takes 5 minutes).

*If signal to noise is still too low you can increase the number of scans **ns** (eg 2X-4X's more) and type **go** to continue signal averaging onto the previous FID.

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. Ok to create directory if you like.

Additional experiments can be collected in a very similar fashion (please see the end of this document). All experiments are easily setup using the 'rpar' command.

<u>Acquisition of DEPT-135 13C NMR spectrum (CH3/CH up; CH2 Down)</u>
edc – create expt 3
rpar dept135.bbo all
ii
zg (experiment takes 5 minutes).

Manually phase from above. Note some peaks are supposed to point down if present.
setti -include appropriate title
pps – autopick peaks
export – save as *whatevername.jpg* and so on. Ok to create directory if you like.

<u>Acquisition of DEPT-90 13C NMR spectrum (CH only up)</u>
edc – create expt 4
rpar dept90.bbo all
ii
zg (experiment takes 5 minutes).

Manually phase from above.
setti -include appropriate title
pps – autopick peaks
export – save as *whatevername.jpg* and so on. Ok to create directory if you like.

<u>Acquisition of DEPT-45 13C NMR spectrum (CH3/CH2/CH all up)</u>
edc – create expt 5
rpar dept45.bbo all
ii
zg

Manually phase from above. All peaks point up.
setti -include appropriate title
pps – autopick peaks

export – save as *whatevername.jpg* and so on. Ok to create directory if you like.
Export your data..

2D HMQC (1H/13C) Correlation via 1 Bond (direct attachment)
edc – create expt 6
rpar hmqc.bbo all
ii
1 td 128
zg

2D processing (for cosy and hmqc above)
xfb – does 2d fourier transform and phase correction.

*scaling of intensities/contours down just like with the 1Ds.
*use LMB and box in area of zoom if you like.
export – save as *whatevername.jpg*

2D COSY (1H/1H) Correlation
edc – create expt 7
rpar cosy.bbo all
ii
zg

2D processing (for cosy and hmqc above)
xfb – does 2d fourier transform and phase correction.

*scaling of intensities/contours down just like with the 1Ds.
*use LMB and box in area of zoom if you like.
export – save as *whatevername.jpg*

When finished email yourself the data

*data .jpgs are stored on desktop

Finishing up with TOPSPIN and logging off the computer

36. Remove your sample and replace it with the CDCl3 standard following steps 3, 4 and 5 above again.

37. Type **exit** to leave the NMR program.

38. logout icon (> type arrow) is on top bar.

39. **rpar** (will give a list of all the expts available. You should focus only ones in **lower case** as expts in ALL CAPS are from standard bruker files). These however can be used too with **prosol** setup methods and please let me know if you'd like to try.