PSU NMR Facility
Acquiring an ^{1}H NMR spectrum
(Quick instructions for Topspin 1.3, Bruker DRX-400)

If you need assistance, please contact:

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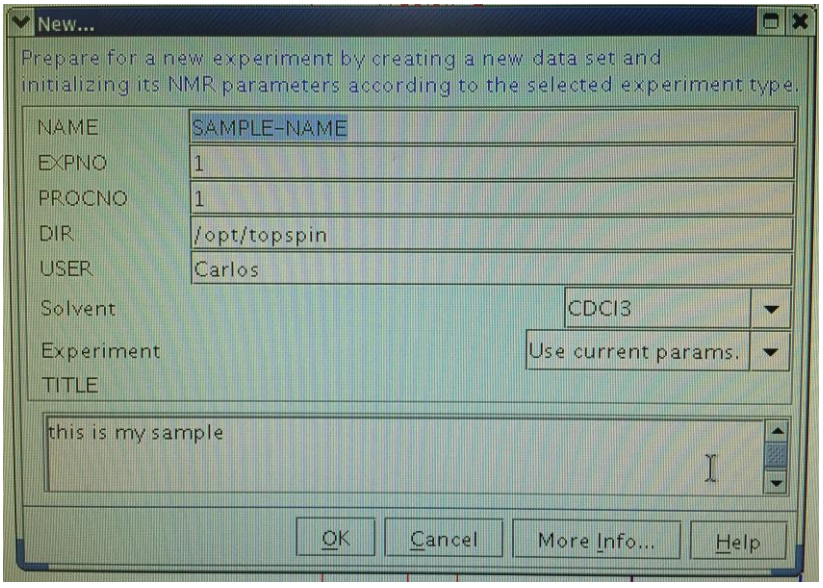
Place your sample in the spinner and carefully adjust the NMR tube depth in the gauge.

1) Turn on the lift air using BSMS (or type ***ej*** <*enter*> at the command line).
   a. If BSMS window is missing, type ***bsmsdisp*** <*enter*> at the command line.

   b. Always wait for the sound of the lift air at full power before placing your sample on top of the magnet!!!

2) Turn off lift air from BSMS (or type ***ij*** <*enter*> at the command line).

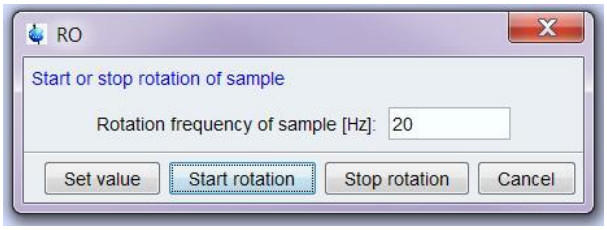
3) Create a new data set by typing ***new*** <*enter*> at the command line or click on  at the toolbar (or type ***edc <enter>*** at the command line)



NAME -- (filename for your compound)
EXPNO -- suggestion: Set 1 for a ^{1}H, 2 for a ^{13}C
PROCNO -- suggestion: set to 1
DIR -- set to /opt/topspin/
USER -- should be <**Your_Professor last name**>
SOLVENT -- your deuterated solvent from the pull-down menu
EXPERIMENT -- "Use current params"
TITLE – the title of your sample (can be as descriptive as needed)

Read a good shimfile with ***rsh***<*enter*>: choose the **stdshims** file from the list, which is the standard shimfile name for all NMR spectrometers.

4) Spin the sample at 20 Hz: type ***ro*** <*enter*> at the command line and click on **Start rotation** at the window:



5) Type ***lock*** <enter> at the command line and choose the appropriate solvent from the lock table.

6) If the lock window is missing, type ***lockdisp*** <enter> at the command line

7) After the lock process is finished, shim **z1** and **z2**.

8) Type ***rpar PROTON all*** <*enter*> at the command line.

9) Type ***getprosol*** <*enter*> at the command line.

   a. For instruments running **Xwinnmr,** type ***eda*** <*enter*> and set prosol to ***TRUE***.

10) Type ***rga*** <*enter*> at the command line

   a. Adjust the number of scans - **NS** - as appropriate if necessary; default for ^{1}H is 16, for ^{13}C is 1024).

11) Type ***zg*** <*enter*> at the command line to start the experiment.

12) For instruments running **Xwinnmr,** type ***acqu*** <*enter*> at the command line if you do not see your FID (raw NMR data).

13) Your NMR data is automatically saved **at the end** of the experiment.

DATA PROCESSING

It is highly recommended to carry out NMR data processing on the *off-line* PC at the NMR Facility, room #10, where you can also back your NMR data up

****Bruker is now offering Topspin software free for Academia: the software can be downloaded at:**
https://www.bruker.com/service/support-upgrades/software-downloads/nmr/free-topspin-processing/free-topspin-download.html

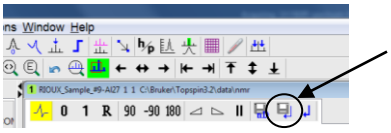
Optionally, you may use the MNova software (the NMR Facility has 50 licenses) – consult the NMR staff to obtain your license and instructions for downloading and activation

14) Type **ef; apk** <*enter*> at the command line to perform Apodization (**e**), Fourier (**f**) Transformation, and automatic phase adjustment (**apk**).

15) If needed, further optimize the phase of the spectrum manually, **using the phase correction icon**.

Click on the phase icon: 

Left-click **0**, hold the mouse button to correct phase **at** the vertical cursor.
Left-click **1**, hold the mouse button to correct phase **away** from the cursor.
Click on the SAVE icon to exit the phasing window.

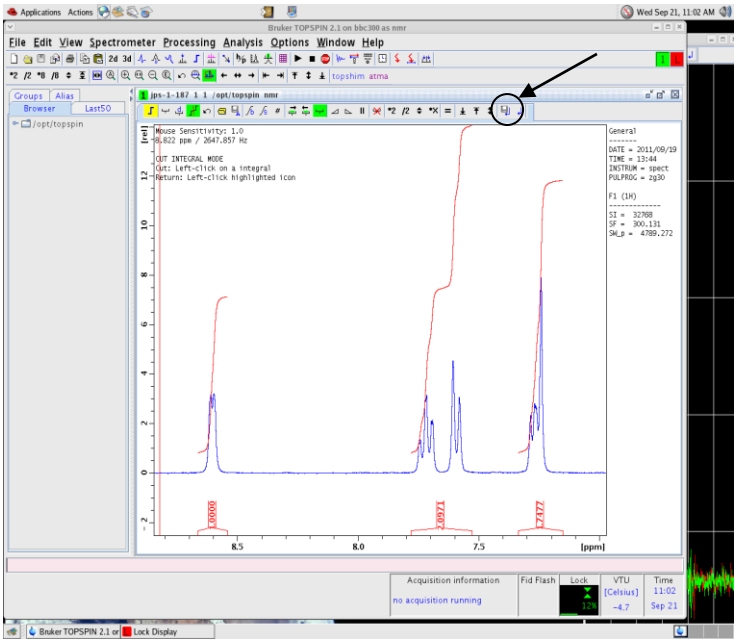


16) Calibrate your spectrum using a known chemical shift (residual solvent peak or TMS). Click on the icon  at the toolbar. Set the reference and return.

17) Type **abs** to flatten the baseline. There is a manual baseline flattening: 

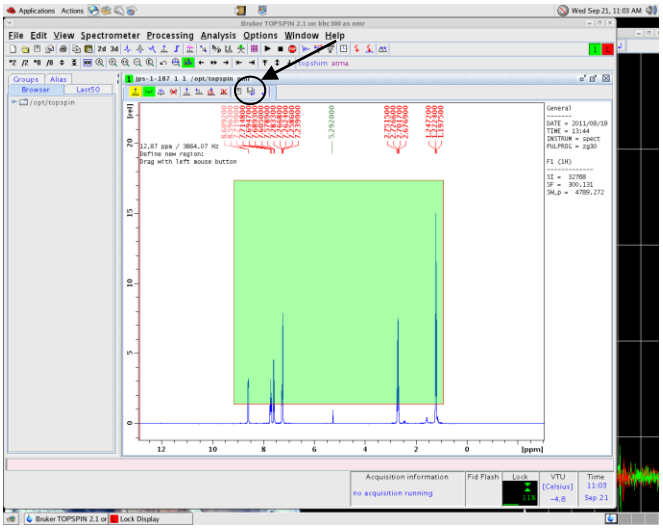
18) Integrate the spectrum manually with  -- This takes you into a subroutine:

Integrate the spectrum, save the integrals before exit the window.



19) Peak picking can be done with the icon  and drawing a green box around the peaks

Save the peaks before exiting the window (using the same icon as the phasing and integration).



20) Type ***plot*** <enter> to open the plot editor (for instruments running TopSpin) and then print.

    a. For instruments running Xwinnmr type ***view*** <*enter*> to print preview and type ***plot*** <*enter*>.

21) Print.

22) Save your data on the PC at Room #10

    a. Save your NMR data **ALSO** on a thumb drive (recommended).

        i. Email the NMR data (as .zip file) is also possible at the off-line PC.