NJIT NMR Lab
Acquiring an NMR spectrum
(Quick instructions for Topspin 1.3, Bruker AV-300)

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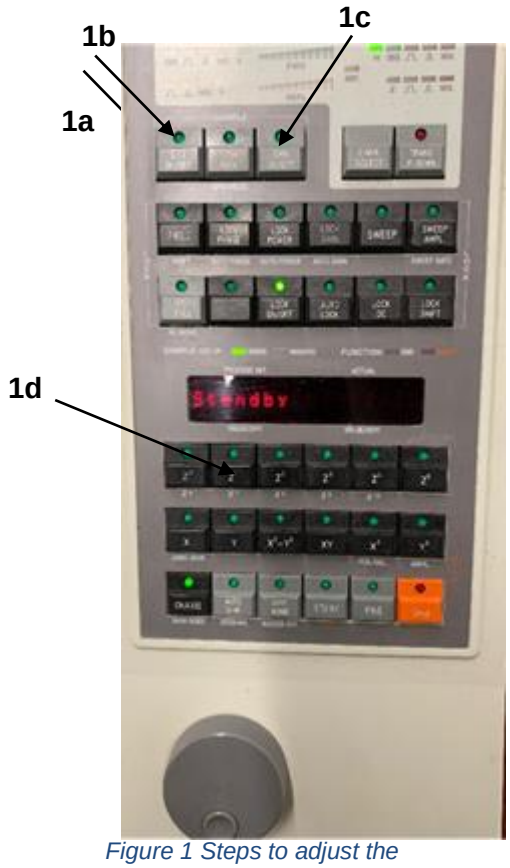


Figure 1 Steps to adjust the spectrometer for NMR acquisition

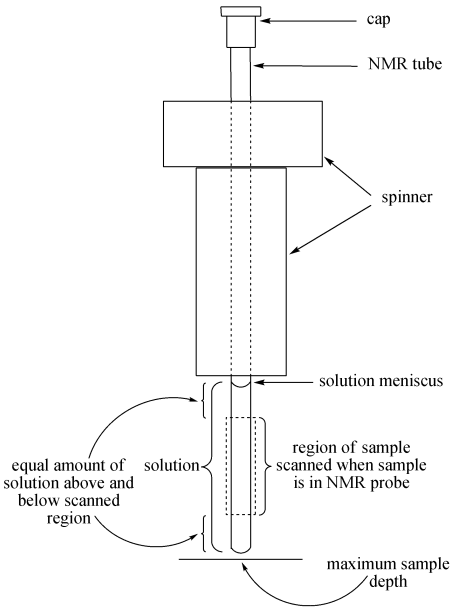


Figure 2 - Adjustment of the position of the NMR tube inside the spinner with the gauge

1) You will use the external BSMS (**B**ruker **S**ample **M**anagement **S**ystem). Follow the arrows in Figure 1.

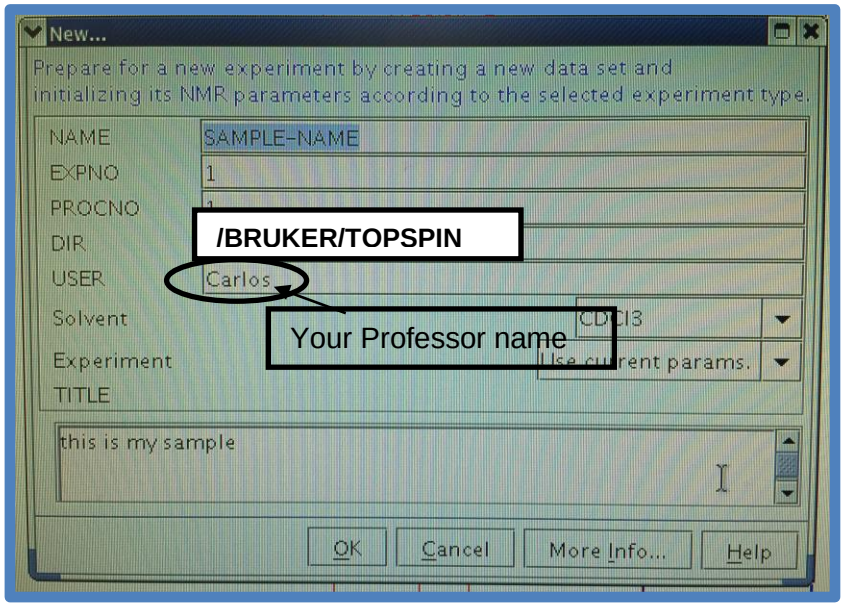
a. Press **button LIFT ON/OFF** (1a) to lift the "dummy" sample that is always being kept inside the probe. Check if the lid is capping the probe: remove it prior to press **button** 1a;

b. Place your sample in the spinner and carefully adjust the NMR tube depth in the gauge (see Figure 2).

⇨ Always listen to the sound of the lift air at full power before placing your sample on top of magnet

c. Press **button LIFT ON/OFF** (1b) to insert your sample into the magnet

2) 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 to start from an existent template)



NAME -- (filename for your compound)
EXPNO -- suggestion: Set 1 for 1H, 2 for a 13C
PROCNO -- suggestion: set to 1
DIR -- set to /BRUKER/TOPSPIN/
USER -- should be <Your_Professor 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)

3) Spin the sample at 25 Hz: press the button shown in arrow 1c in Figure 1.

4) Read a good shimfile with rsh <***enter***>: Choose the **stdshims_CP-300** file from the list, which is the standard shimfile name for the NMR spectrometers.

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

6) After the lock process is finished, shim **z1, z2, z3, and z4** pressing the buttons shown in Figure 1, arrow 1d.

7) Type ***rga*** <*enter*> at the TopSpin command line.

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

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

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

10) Finishing the experiment, press lift button -1a- to eject your sample and insert the "dummy" sample, pressing the lift button again, 1b. Cover the probe with the lid and lock on the "dummy" sample (D_2O).

DATA PROCESSING

It is highly recommended to carry out NMR data processing off-*line*

****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 (NJIT's NMR laboratory will have 5 licenses) -email me to obtain your license and instructions for downloading and activation

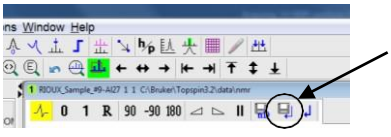
Processing using TopSpin

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

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

- Click on the phase icon: 

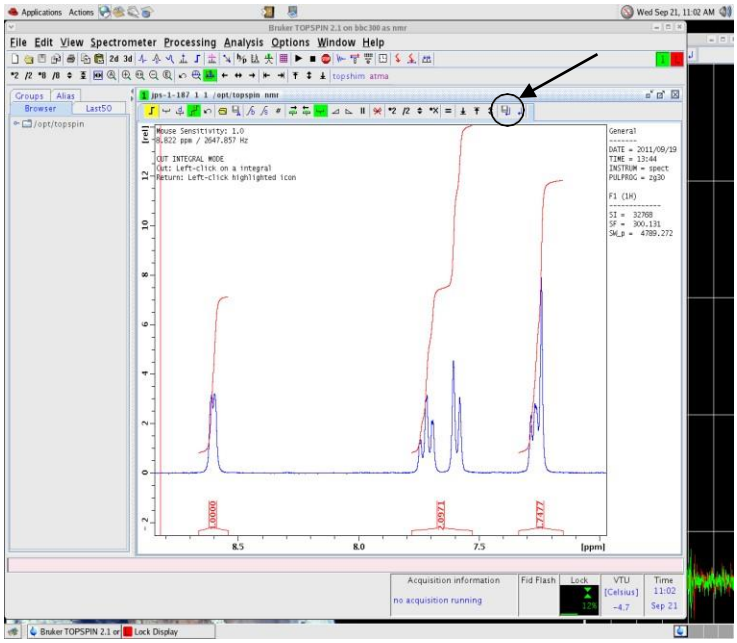
Left-click **0**, hold the mouse button to correct phase **at** the vertical cursor. Left-click **1**, hold the mouse button to correct phase **away** from the cursor. Click on the SAVE icon to exit the phasing window.



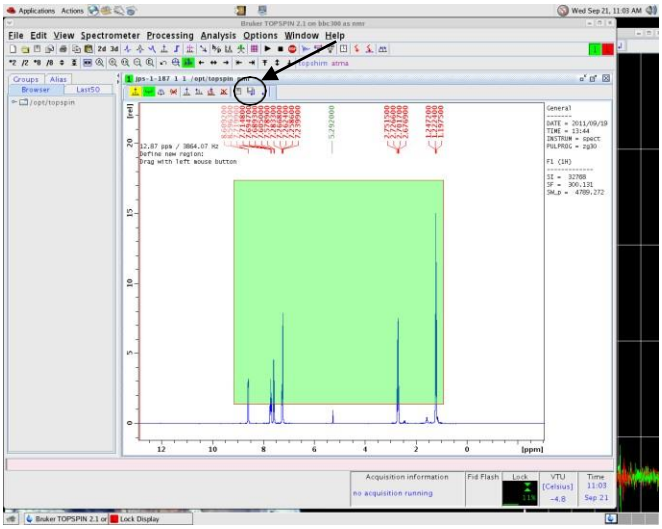
- Calibrate your spectrum using a known chemical shift (residual solvent peak or TMS). Click on the icon  at the toolbar. Set the reference and return.

- Type **abs n** to flatten the baseline. There is a manual baseline flattening: 

- Integrate the spectrum manually with  -- This takes you into a subroutine: Integrate the spectrum, save the integrals before exit the window.



- 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).



- Save your data on the flash drive (alternatively, zip the NMR data directory and email to you as attachment).