NJIT NMR Lab

Acquiring an NMR spectrum

(Quick instructions for Topspin 1.3, Bruker AV-300)

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Instructions for H1 NMR ONLY

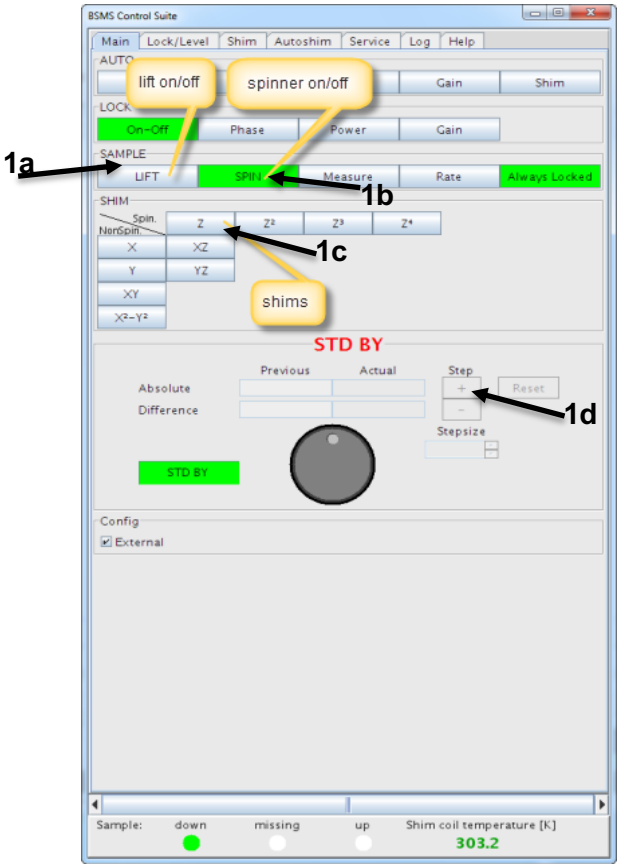


Figure 1: Steps to Adjust the Spectrometer for NMR Acquisition

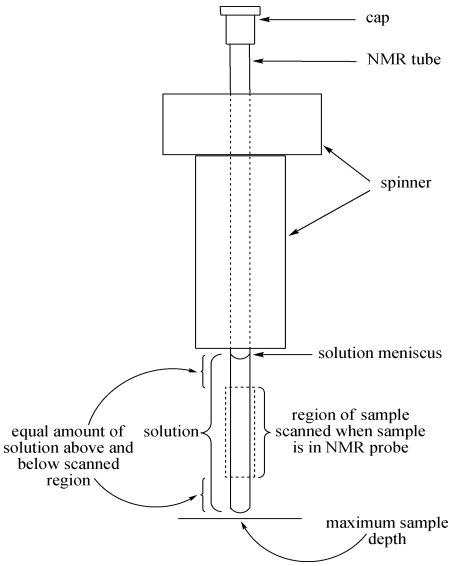


Figure 2 - Adjustment of the position of the NMR tube inside the spinner with the gauge

1) Use the **INTERNAL** BSMS (**B**ruker **S**ample **M**anagement **S**ystem). Follow the arrows in Figure 1.

2) **Type on TopSpin command line: BSMSDISP <enter>**

   a. Press the **LIFT** (1a) to lift the "dummy" sample that is always kept inside the probe (**locked on DMSO-d6**). Check if the lid is capping the probe: remove it before pressing **button** 1a.

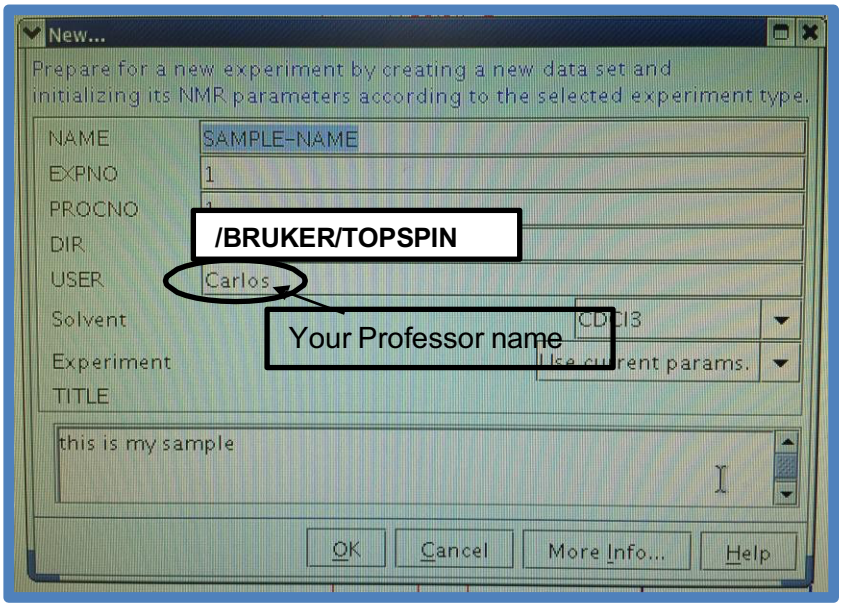
   b. Place your sample in the spinner and carefully adjust the NMR tube depth in the gauge (see Figure 2).

c. Place your sample on top of the magnet, ensuring it is floating.

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

d. Press the **LIFT** (1a) button **again** to insert your sample into the magnet.

3) Create a new data set by typing **new** <<*enter*> at the command line or clicking on  the toolbar (or typing **edc** <*enter*> at the command line to start from an existent template).



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

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

6) Type **WOBB <enter>** at the TopSpin command line, and a display, such as Figure 3, will appear. Rotate the monitor, facing it now with the 300-MHz magnet.

7) Go to the PROBE and adjust the **YELLOW** capacitor wand marked **'T'** with the red tool at the base of the magnet. Rotate this capacitor wand and look at the monitor, moving the dip **towards the center (see the RED vertical line).**

8) Once the dip is centered, rotate the monitor back, and type **stop <enter>** at the TopSpin command line.

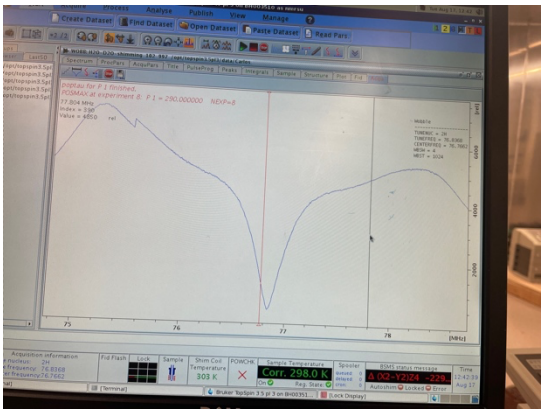


Figure 3

9) Spin the sample at 25 Hz: press the button shown in arrow **1b** in Figure 1.

10) Select a suitable shimfile using rsh <*enter*>: Choose the **stdshims_CP-300** file from the list, as it is the standard shimfile name for the NMR spectrometers.

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

12) After completing the locking process, shim **z1, z2, z3, and z4 by** pressing the buttons indicated in Figure 1, arrows **1c and 1d**.
a**. 1d are the + or - buttons that should always be selected, always observing the lock level on the lock display. The goal is to maximize the lock level to obtain the best homogeneity.**

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

a. Adjust the number of scans (NS) as necessary; the default is 16 for 1H and 1024 for 13C. Type' zg' and press ***enter*** at the command line to start the experiment.

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

15) After finishing the experiment, press the 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 that NMR data processing be carried out off*line*.

****Bruker is now offering Topspin software free for Academia: the software can be downloaded at:** https://www.bruker.com/protected/en/services/software-downloads/nmr/software/topspin37.html

You may optionally use the MNova software (NJIT's NMR laboratory will have five licenses). Email me to obtain your license and instructions for downloading and activation.

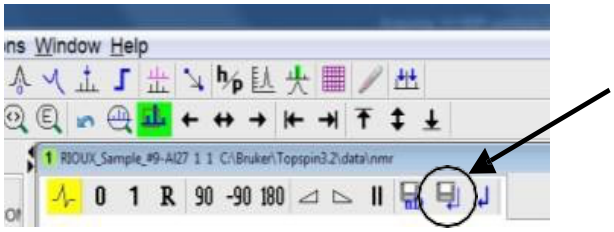
Processing using TopSpin

- 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 AND hold** the mouse button to correct the phase **<u>at</u>** the vertical cursor. Left-click **1 AND hold** the mouse button to correct the phase **<u>away</u>** from the cursor. Click the SAVE icon to exit the phasing window.

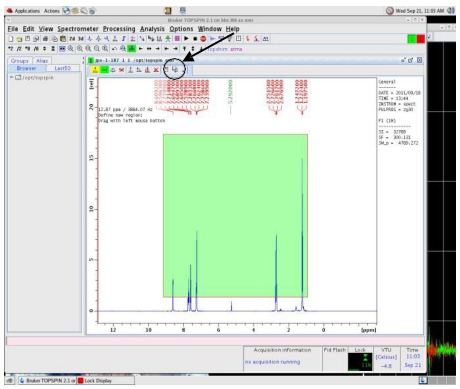


- Calibrate your spectrum using a known chemical shift (residual solvent peak or TMS). Click on the icon  in the toolbar. Set the reference and save it with the icon pointed in the figure above.

- Type' **abs n'** to flatten the baseline. There is a manual method for flattening the baseline. 

- **<u>Integrate</u>** the spectrum manually. This will guide you further into a subroutine: integrate the spectrum and save the integrals before exiting the window.

- **<u>Peak picking</u>** can be done using the icon.  and drawing a green box around the peaks. The peaks can be saved before exiting the window by using the same icon as for phasing and integration.



Save your data to the flash drive, or alternatively, compress the NMR data directory into a zip file