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

If you need assistance, please contact: Dr. Carlos Pacheco -- Office # Tiernan B-006 - <u>carlos.n.pacheco@njit.edu</u>; Ph# (office): 973-596-6534; Ph# (lab): 973-596-6551; cell: 609-240-5957



- 1) You will use the external BSMS (Bruker Sample Management System). 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)

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PROCNO DIR	/BRUKER/TOPSPIN		
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Solvent Experiment	Your Professor name Use current params.		

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 <u>at the end</u> 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/freetopspin-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: -1-



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). at the toolbar. Set the reference and return. Click on the icon
- 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.

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- 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).

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