# Measuring 1H NMR Spectra

(This guide is intended for routine use on the 300MHz spectrometer)

1. First login as a user in the Linux workstation. If you don't have an account please contact us to setup one for you. Choose your username and type the password.
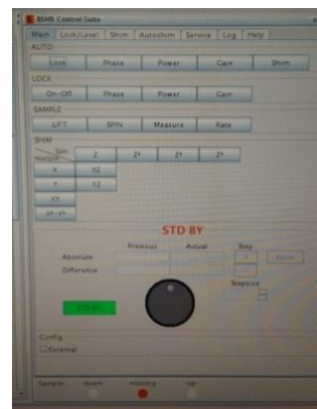
2. Double click the topspin icon

3. Open a dataset by clicking the Browser menu from the left-hand side panel and choosing appropriate data set. The users' datasets are stored in /opt/topspin3.2pl7. To open a data set listed in the browser double click or drag and drop. Type edc to create a new file, and enter the parameters: experiment name in the field NAME, experiment number in EXPNO (is changed if there are already experiments under the same name) and the title in TITLE (The title can be changed later by clicking on the TITLE tab on the spectrum window). Click on OK to create the file.

4. Inserting Sample: NMR tube containing the sample should be held in a blue spinner. Place the NMR tube in the spinner and the spinner in the sample gauge. Push the sample tube so that the depth of the sample above and below the center line of the sample depth gauge is equal. However, never exceed the lower limit as this can damage the probe as well as the sample.

5. Click on (or type *bsmsdisp*) to open the BSMS display window. Press the Main tab where most of the required actions are displayed. By clicking on the LIFT button in the SAMPLE frame, air is passed through the magnet. It is important to wait for the airflow and only then you can put the sample in the magnet. Click on LIFT button again to stop the airflow and the sample drops into the magnet. After the bsmsdisp shows that the sample is

inserted successfully (a green light appears down) you can click on SPIN to spin the sample (wait until the red light is replaced by a green one).

6. Locking: Lock signal can be seen in the lock display window that can be opened by typing *lockdisp* at the command line or by clicking on [icon] . Enter lock at the command line and select the appropriate solvent from the solvent list and wait for the "lock finished" message.

7. Shimming: To read the shim file type *rsh* and select a recent file (written on a note next to the screen).

In the *bsmsdisp* window under the Main tab in the SHIM frame there are buttons for the main shim functions. Choose the shim function that you want and change it by scrolling the mouse wheel or by clicking on Step + and Step – in the VALUE frame. If the sample is spinning then only the **Z** axis shims (**Z**, **Z2**, **Z3**) are adjusted, if not the other shims also need to be adjusted. The higher the signal in the *lockdisp* window, the better the shimming is. If the lock signal exceeds the lock display window, lower the GAIN value (on the LOCK row) in the *bsmsdisp* window. When shimming is done press STDBY.

8. Adjust the sensitivity of the ADC by typing *rga*, wait for the "rga finished" message.

9. To start acquisition type *zg*. After a while type *tr* to transfer data to processing and *efp* to perform Fourier transform.

If needed increase the number of scans (ns), which will improve the signal-to-noise ratio of the spectrum, but the total experiment time will also be increased. After a sufficient signal-to-noise ratio is obtained the experiment can be stopped by typing *halt* and *efp* for processing the obtained data.

10. Processing:

Type *apk* (automatic phase correction) to phase the spectrum.

Manual phase correction can be done by pressing "Process" and "Adjust phase". A vertical red line appears and by left clicking on **0** and dragging the mouse up and down the zero-order phase is corrected. First order phase correction (far from the red vertical line) is done by left clicking **1** and dragging the mouse up and down.

When the phase correction is complete click on [icon] to save (or on [icon] to cancel) the correction.

The spectrum may be expanded by dragging the mouse while left-clicked. Releasing the mouse key expands the spectrum.

For Baseline correction type *absn.*

Peak peaking: type *ppf* for automatic peak picking. The minimum intensity can be adjusted by changing *MI* and then type *ppf* again.

Integration: enter *.int* or click on "Integrate" in the "Process" tab. The integration window will open. You can add an integral by clicking on and left dragging the cursor over the regions of the spectrum that you want to integrate. Calibrate the integral intensity by right clicking on an integral of known intensity (of a known number of protons). A menu will appear; select *Calibrate current integral* and enter the intensity in the New value field. Click on [icon] to save the integration.

Print: click on the printer button [icon] and three times enter.

11. When finishing work remove the sample: In the *bsmsdisp* window click on SPIN to stop spinning (green light turns gray), LOCK On-Off to unlock the sample (green light turns gray) and then click LIFT so the airflow ejects the sample. Remove the sample and click LIFT again to turn off the airflow.
Close the TOPSPIN window.

Leave your account: Menu > leave (don't press leave since this will turn off the computer) > log out.