HOW TO RESERVE TIME:

The sign up calendar for the 300 MHz spectrometer is located next to the printer in the NMR room McGowan South 300. Attempts should be made to reserve time in advance. If the experiment to be conducted will take more than 30 minutes, it is best to wait until the end of the work day and run the experiment in the evening or overnight.

LOGGING ON:

Log onto Windows using the user name of the group or class you are working on. For Research use the **Research Student User** account, for classes use **Teaching Lab User** account. Passwords are obtained once access has been granted to the NMR instrument and they are retained on file by Massimo Pacilli in AJM-317.

OPEN TOPSPIN SOFTWARE:

- Double click on **TopSpin 1.3** icon.
- Click on **New** under the **File** tab and fill the field as follows:

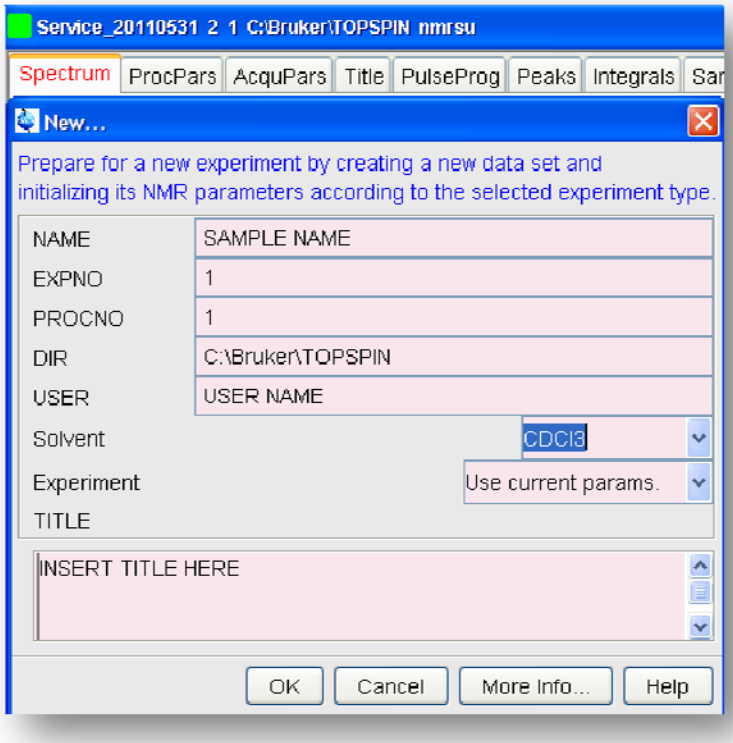

Figure 1. New Window in TopSpin.

- Click on **OK** button.
- Click on **BSMS** and **LOCK** buttons in the toolbar in order to open the lock window, and BSMS Panel.

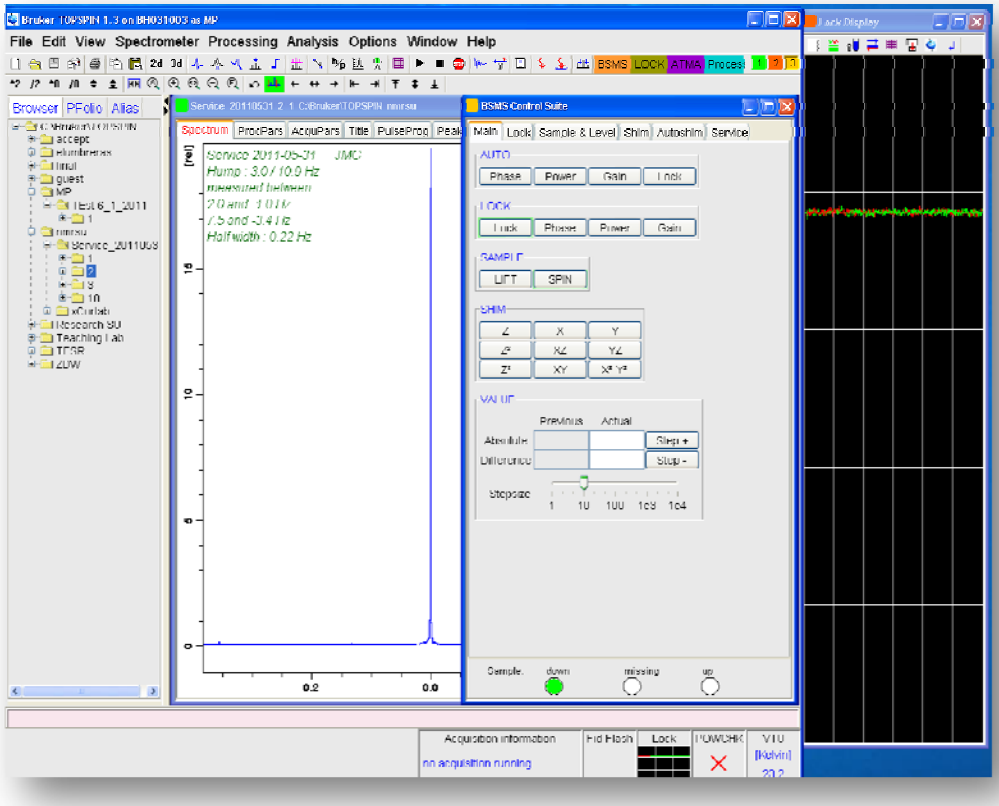

Figure 2. Lock and BSMS Panels.

<u>INSERT THE SAMPLE TO BE ANALYZED:</u>

- In the BSMS panel: click on **SPIN** and **LOCK** buttons in order to turn off spin and lock, then click on **LIFT** in order to bring the standard sample out of the probe.
- Remove the standard sample without touching the sides of the blue spinner.
- Replace the sample to be analyzed in the spinner and slide to the premeasured depth (5mm) using the depth gauge. Place the sample in the magnet resting on the air stream.
- Click on the **LIFT** button to lower the sample into the instrument and click on **SPIN**.
- Make sure the experiment window is active (not the BSMS one). Type **lock** in the pink command line in the bottom toolbar and select the appropriate solvent used and click **OK**.
- Wait for signal to lock, both LOCK and SPIN signal should be highlighted at this point.

SHIMMING:

- Click on the **Z** and **Z2** buttons on the **BSMS** display to adjust the **Z** and **Z2** shims in order to optimize the signal by minimizing the noise of the lock signal.
- Click with the left mouse button on **Z**. The button will be highlighted, indicating it is active.
- Place the cursor along the slide bar below the buttons to control the step size by moving the slide bar (shim at steps 10, 5, 2).
- Increase the shim value by clicking on the Step + button, in order to decrease it click on the Step - button. Adjust until the lock signal reaches its maximum value.
- Click with the left mouse button on **Z2** and repeat to reach a new maximum.
- Click again on the **Z** and proceed to optimize the shim. This process is iterative; you may continue adjusting the shim toggling back and forth between Z and Z2 until desire shim is obtained.
- If the lock signal raises above the top of the lock window, it can be brought back to scale by clicking on **GAIN** button under the LOCK tab and left clicking on the lock window to bring the signal to desired level.

SETTING UP THE EXPERIMENT:

- Type **rpar** to bring up experiment menu (i.e. proton, carbon etc…) and click on **copy all** for the desired experiment.
- Type **ns** to change the number of scans.
- Type **getprosol** to initialize instrument.

COLLECTING THE DATA:

- Type **atma** to automatically tune and match the probe, then wait until the prompt says: **atma: finished**.
- Type **rga** to automatically set the receiver gain, then wait until the prompt says: **rga: finished**.
- Type **zg** to start acquisition.
- When the acquisition is complete the following message will appear: **checklockshift: Finished**.
- If necessary, you may stop the acquisition by typing **halt**. This will interrupt the experiment while still saving the data already acquired.

PROCESSING THE DATA:

- Type **bc** in order to apply a baseline correction; type **ef** in order to apply exponential multiplication and Fourier transformation; type **apk** to adjust the phase of the peaks and **.all** to bring the spectrum to scale. Alternatively pressing the **PROCESS** button in the toolbar will carry out all of these commands in sequence.
- Set the TMS reference to 0 ppm (or any other peak to a given value) by clicking on the spectrum calibration icon on the top toolbar. Align the red cursor with the TMS and click to set the spectrum calibration frequency to 0 ppm.
- Click on the **Integrate** icon on the topspin toolbar. A new toolbar will appear above the spectrum, which will allow you to **Define Integrals**. After selecting the peaks, you may **Calibrate** a peak to a given value, **Normalize** the sum of the integrals to a given value, or **Delete** the integrals selected.
- When integration is complete, click the **Save and Return** icon.

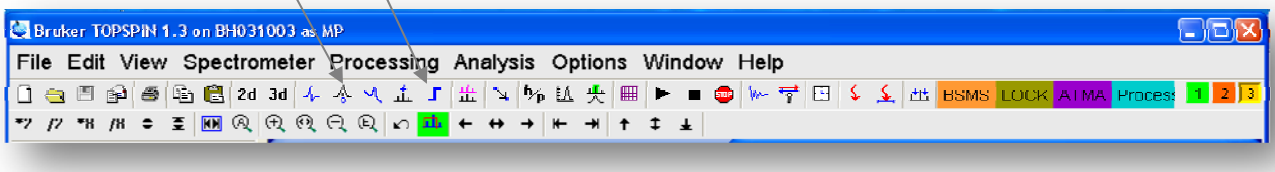

Figure 3. TopSpin Toolbar.

PRINTING THE SPECTRUM:

- Choose the **autoplot** option under **Print** in the **File** and the spectrum will print as it appear on the screen.
- Additional adjustments can be made by typing **xwinplot**. In this mode it is possible to expand region of the spectrum, insert zoomed areas and access other features by navigating the menu screen, which is accessed by right clicking on the spectrum and left clicking on **1D/2D Edit**.

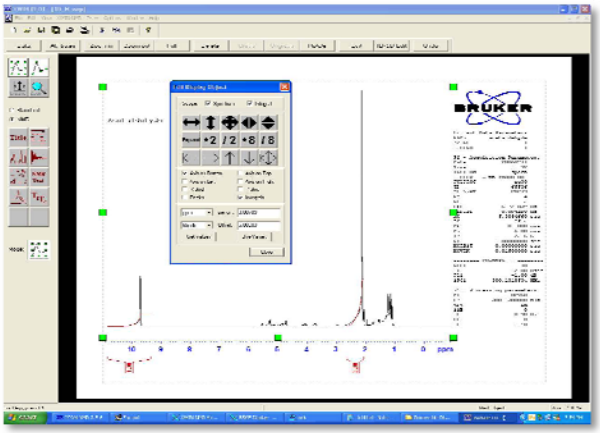

Figure 4. Xwin Plot Editing Mode.

OBTAINING A PEAK LIST:

- You may toggle between Hz and ppm units by clicking on the **h/p** icon in the toolbar.
- In order to display peaks on the spectrum or obtain a peak list, click on the **Peak Picking** Icon and a new tool bar will appear. You may define peak picking ranges and modify selections by navigating the toolbar above the spectrum.
- Select the area of interest by clicking and dragging the cursor on the spectrum. Right-Click on the spectrum in this mode in order to obtain the peak list.

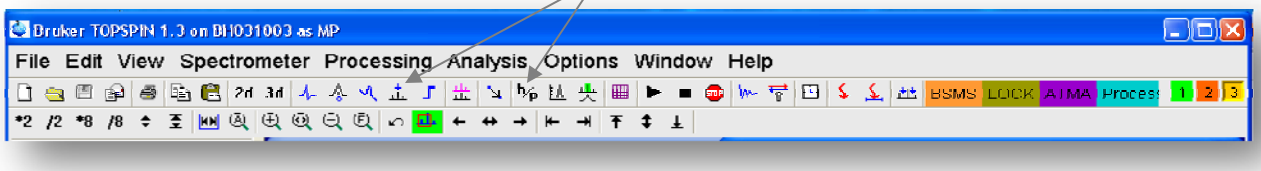

Figure 5. TopSpin Toolbar.

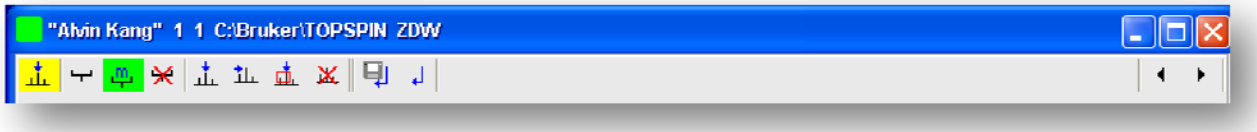

Figure 6. Peak Picking Toolbar.

SHUTDOWN and LOG OUT:

- Click on **SPIN** and **LOCK** buttons on the BSMS panel.
- Eject sample by clicking on **LIFT**.
- Replace the sample with the standard acetone sample.
- Proceed to **SPIN** button and type **Lock** in the pink command line. Select acetone as the solvent for the standard.
- In order to shim the standard type: **rsh** in command prompt and select **mp_latest** from the list.
- Close the shim and lock windows, exit program and log off.