

To Setup Experiment.

1. Click on the **'Start'** tab in the TopSpin Menu bar.
2. Select **'create dataset'** by clicking on it.
3. Enter/Choose the following information in to the 'New' window
 - a. Name (of your experiment)
 - b. Experiment (standard options are listed below)
 - i. Standard ^1H = type or choose **'PROTON'**
 - ii. Standard ^{13}C = type or choose **'C13CPD'** (to adjust # of scans (NS) choose **'AcquPars'**, set NS=512 for 30 min acquisition time, 1028 for ~1 hours, etc)
 - iii. ^{31}P observed = type or choose **'P31'**
 - iv. ^{31}P Proton decoupled = type or choose **'P31CPD'**
 - c. Directory (choose your Pls name)
 - d. Title

***DO NOT change any other parameters.**
4. Click 'OK' in the 'New' window.
5. Click on the **'Acquire'** tab in the TopSpin menu bar.
6. Select **'Sample'** by clicking on it.
7. Select **'ej'** by clicking on this option (this ejects the sample).
8. Place the spinner with the reference sample into the depth finder, replace the reference tube with your sample, and then place the sample+spinner on the top of the magnet.
9. Select **'ij'** by clicking on it (this injects the sample).
10. Select **'lock'** by clicking on it. A menu of solvents pops up.

Choose the solvent and click 'OK' – wait till you see *lockn: finished* at the bottom of the screen
11. Select **'Tune'** by clicking on it.

Wait for the instrument to tune; wait till you see *atma: finished* (bottom left of the screen)
12. Select **'Spin'** by clicking on it.

Select **'Turn sample rotation on'** to turn sample spin on.
13. Select **'Shim'** by clicking on it and wait for the process to complete (*topshim: completed*).
14. Select **'Prosol'** by clicking on it and wait for the process to complete (*prosol: finished*).
15. Select **'Gain'** by clicking on it and wait for the process to complete (*rga: finished*).
16. To start your acquisition, press **'Go'**.

Data Processing.

1. Click on the **'Process'** tab in the TopSpin Menu bar.
2. Click on **'Proc Spectrum'**. This executes a Fourier transform, automatic phase and baseline correction.
3. Use the pick peaks tab to label your peaks. When finished click on **return & save**.
4. Use the integrate tab to integrate your peaks. When finished click on **return & save**.

Set the cursor line, starting at the left of the spectrum, to the left of the first peak to be integrated, click the left mouse button and drag the cursor line to the right of the peak, then release the mouse button.
5. To plot data click on **'Plot'** and modify the layout as you see fit.
6. Click on the printer icon in the left top corner to plot the spectrum.

When you are finished.

1. Click on the **'Acquire'** tab in the TopSpin menu bar, and select **'Sample'** by clicking on it.
2. Select **'ej'** by clicking on this option (this ejects the sample).
3. Place the spinner+your sample into the depth finder, remove your sample and then place the reference (sealed tube) into the spinner and put it on the top of the magnet.
4. Select **'ij'** by clicking on it (this injects the sample).
5. Close the window of your experiment! Don't close the TopSpin window!!
6. Log your experiment(s) into the log book