Bruker Fourier300 NMR Spectrometer

Sample solution height must be 4.5 cm with sample fully dissolved in a deuterated solvent.

Open Bruker TopSpin 3.2 software. (Icon on desktop.)

Defining Sample

1. Type "**new**" and enter information for your sample:
   NAME – **Folder name** (Your last name would be good). This will create a new folder for your dataset, or you may add to an existing folder by typing its name here.
   EXPNO – Increment this number for each sample you run.
   PROCNO – This value must remain "1".
   Experiment – for ^{1}H, use "**PROTON**"; for ^{13}C, use "**C13CPD**"; for other options click "Select".
   Set solvent – choose appropriate solvent from drop down menu.
   Leave " Execute getprsol" and "Keep Parameters" unchecked. Leave "Receivers" set to 1.
   DIR- Make sure the directory is set to "C:\Bruker\TopSpin3.2\data\nmrsu\nmr\Chem250"
   TITLE- Enter experimental details or the **sample name**.
2. Click **OK**.

Inserting Sample (Check that dust cap has been removed.)

1. Type "**ej**" to eject previous sample from NMR. Remove old sample from spinner, and carefully place your sample into the spinner. Use plastic depth gauge to check appropriate solution height: push tube through spinner gently to the bottom of gauge. Put your spinner and tube into the NMR sample probe. It should float.
2. Type "**ij**" to lower the sample into the probe. You will hear a click when sample is properly positioned in probe.

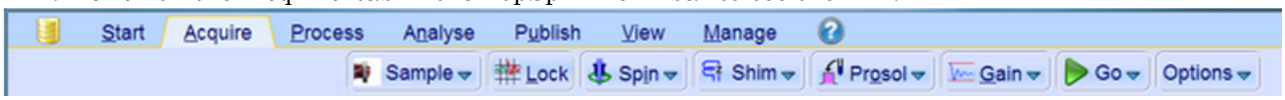
Locking and Shimming

1. Type "**lock**". The "Solvents Table" window will pop up. Choose your solvent to lock on. *Watch progress at bottom left.* Wait for "lockn: finished" but message disappears if move cursor.

2. Type "**ro on**" to spin the sample. *Watch progress at bottom left.*
3. Option: type "**gradshim**" or click on "Start Gradient Shimming". *Watch progress at bottom.*
4. Type "**getprosol**" to get standard acquisition parameters. *Watch progress at bottom left.*
5. Option: Type "**ased**" to modify parameters such as number of scans (ns) or delay (D1).
6. Type "**rga**" to set the receiver gain.

Collecting Data

1. Type "**xaua**". *Watch progress at bottom left.*
2. Click on the 'Acquire' tab in the TopSpin menu bar to see the FID.



3. You can check if you have enough data by typing "tr" and then processing as listed below. If enough samples have been acquired, type 'halt' to save all scans acquired and stop acquisition.

Processing Spectra

1. Click on the 'Process' tab in the TopSpin menu bar.
2. Select 'Proc. Spectrum' button to load, Fourier transform, and phase your data or type "efp", then "apk", then "abs". For manual phase adjustment so that peaks are symmetric and go up, type ".ph". Right click at one end of the spectrum and select 'Set Pivot Point'. Click and drag the '0' in the upper left to adjust peaks near the pivot point; click and drag the '1' to adjust the rest.
3. Type "**pp**" or select 'Pick Peaks' button. Drag a box that includes the top of the peaks. Click on the Save+Return icon to keep values or return icon to exit. Type "sref" to set a specific value.
4. Type "**.int**" or select 'Integrate' button. With the field goal icon yellow, click and drag over a peak to integrate. To set a specific integral value, right click and select 'Calibrate Current Integral'.
5. Click 'Plot' on the spectrum toolbar or type "plot". Set paper settings to 'Letter'. A useful layout file is "Standard KLB.xwp". Zoom range can be specified by typing ".zx" or ".all".

Always store a sample in spectrometer when finished! Type "ro off" and replace dustcap.