

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

- Type “**new**” in the bottom command line and press return. Enter information for your sample:
 NAME – **Folder name** (Your last name would be good). This will create or add to a folder.
 EXPNO – **Increment this number** for each sample you run or your data is overwritten.
 PROCNO – This value must remain “1”.
 Experiment – for ^1H , 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- “C:\Bruker\TopSpin3.2\data\nmrsu\nmr\course\name” from drop down menu.
 TITLE- Enter experimental details or the **sample name**.
- Click **OK**.

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

- 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.
- Type “**ij**” to lower the sample into the probe. You will hear a click when sample is properly positioned in probe.

Locking and Shimming

- Type “**lock**”. The “Solvents Table” window will pop up. Choose your solvent to lock on.
Watch progress at bottom left but moving the cursor clears the message. Wait for “lockn: finished”. At the bottom right a green check after the word locked indicates success. If the lock is way off, try “flock” instead. If the lock window is missing, type “lockdisp”.
- Type “**ro on**” to spin the sample. *Watch progress at bottom left.*
- Option: type “tune” and choose zz2 (the last option) or recall previous shim settings with “rsh”.
 Option: type “**gradshim**” or click on “Start Gradient Shimming”. If you get a good shim, “wsh”.
 Option: type “bsmsdisp” to manually adjust shims (starting with z and z²) by clicking on Step+ or Step- to maximize signal. Spin off before adjusting x or y spins.
- Type “getprosol” to recall standard acquisition parameters. Shimming or nucleus changes values.
 Type “**ns**” to set the number of scans. For samples that dissolve well, 2 or 4 is enough for proton.
 Options: Type “ased” to modify parameters like delay (D1). Type “expt” to see time required.

Collecting Data

- Type “**xaua**”. *Watch progress at bottom left (rga, zg)*
- Click on the **Acquire** tab in the TopSpin menu bar to see the FID.
- 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

- Click on the **Process** tab in the TopSpin menu bar.
- Select **Proc. Spectrum** button to Fourier transform, phase, and reference your data (*ef, apk, sref, abs*). Option: Right click on the spectrum to select Show Full Spectrum.
- When you enter a mode, icons appear at the top of the plot window. Click on the Save+Return icon to keep changes or the Return icon (top right) to exit. 
 Mode: Type “.ph” for manual phase adjustment. Right click at one end of the spectrum and select ‘Set Pivot Point’. Click and drag the ‘0’ icon in the upper left to adjust peaks near the pivot point; click and drag the ‘1’ icon to adjust the rest. Exit mode.
 Mode: Type “.pp” or select *Pick Peaks* button. Exit mode.
 Mode: Type “.int” or select *Integrate* button. With the field goal icon yellow, click and drag over a peak to integrate. To set an integral value, right click and select *Calibrate Current Integral*.
- Click **Plot** on the spectrum toolbar or type “plot”. Paper orientation should be *Landscape*. A useful layout file is “Beloit”. Click on spectrum, “Axes” and *Plot limits x: 12 -.5 (H) or 250 -10 (C)*.

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