
Bruker Fourier300 NMR Spectrometer

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

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

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.

Defining Sample

1. Type “**new**” in the bottom command line and press return.
Enter information for your sample:
NAME – **Folder name** (Your last name). This will create or add to a folder.
EXPNO – **Increment this number** for each sample you run.
PROCNO – This value must remain “1”.
Experiment – for ^1H , use “**PROTON**”; for ^{13}C , use “**C13CPD**” with decoupled protons; for other sequences click “Select”. See table below.
Options
Set solvent – choose appropriate solvent from drop down menu.
Leave “Execute getprsol” and “Keep Parameters” unchecked.
DIR- “C:\Bruker\TopSpin3.2\data\nmrsu\nmr\coursename” from menu.
Leave “Receivers” set to 1.
TITLE- Enter experimental details or the **sample name**.
2. Click **OK**.



Locking and Shimming

1. Type “**lock**”. The “Solvents Table” window will pop up. Choose your lock solvent. The trace will scan back and forth in the lock window at the right. If the lock window is missing, type “lockdisp”. Higher is better.
Check completion at bottom left of main window 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.
2. Type “**ro on**” to spin the sample. *Watch progress at bottom left.*
3. Option: type “**tune**” and choose zz2 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.
4. Type “**getprosol**” to recall pulse width and power levels.

Collecting Data

1. Type “**ns**” to set the number of scans. For samples that dissolve well, 2 or 4 is enough for proton and 238 is a guess for carbon. Options: Type “**ased**” to modify parameters like delay (D1). Type “**expt**” to see time required.
2. Type “**xaua**”. *Watch progress at bottom left (rga, zg)*
3. Click on the **Acquire** tab in the TopSpin menu bar to see the FID.
4. 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 Fourier transform, phase, and reference your data (*ef, apk, sref, abs*). Option: Right click on the spectrum to select Show Full Spectrum.
3. 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. Left click and drag a box that includes peak tops. Save+Return.
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*. Save+Return.
4. 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.

Additional Experiments

C13DEPT45	13C DEPT45, all positive
C13DEPT90	13C DEPT90, CH only
C13DEPT135p	13C DEPT135, CH and CH ₃ positive, CH ₂ negative, phase of previous 13C
C13GD	13C experiment, no decoupling
C13IG	13C experiment, with decoupling, no NOE (inverse gated decoupling)
C13MULT	13C automatic multiplicity determination
C13OFF	13C exp. off resonance
PROHOMODEC	1H homo decoupling experiment
PROTONT1	1H T1 Relaxation measurement
COSYGPSW	two-dimensional 1H gradient
HSQCEDETGP	sw opt. edited HSQC with gradients