

NMR - Recording a ^1H NMR Spectrum — Bruker 400 MHz

NOTE: Only students who have been properly trained in the use of the Bruker 400 and who have signed the User Rules are permitted to use this instrument.

1. Log into Windows and click the Topspin icon.
If you wish to save the data to your “home” drive, click the *Map_H_Drive* icon on the Desktop and enter your Wilkes username and password.
2. *Create a new datafile* by typing EDC.
3. *Insert sample:* EJ to eject the standard; IJ to inject your sample.
4. *Lock:* If your sample is in the same solvent as the previous one, it should lock automatically. If not, type LOCK CDCL3 to lock on the deuterium signal; for other solvents, type LOCK and pick from the menu.
5. *Shim:*
automatic shimming: type TOPSHIM 1DFAST .
manual shimming:
 - i. open the BSMS panel (BSMSDISP or **B** button).
 - ii. adjust the Gain to place the lock level $\frac{3}{4}$ the way up the screen.
 - iii. tweak the Lock Phase to maximize the lock signal.
 - iv. alternately adjust Z, Z², (and Z³) to maximize the lock signal.
decrease the gain as necessary to keep the lock on the screen.
end with Z
6. *Set up the spectrometer:*

RPAR PROTON ALL	read in all ^1H parameters
GETPROSOL	read in probe and solvent parameters
NS 4 ; DS 0	reduce the number of scans
- (7) *Tune probe* if doing a non-H nucleus: ATMA.
8. *Acquire Data:* Return to the main window and type:

RGA	autogain (For ^{13}C and other weak nuclei, type RG 200 instead).
ZG	zero old data and get new data.

⇒ At this point, if you have saved your data to the H-Drive (“home”), you may process it with iNMR. Skip to step 11 below.
9. *Process the data:*

EFP	weight and transform the FID; apply phase correction from previous spectrum (“Exponentially multiply, FT, Phase”)
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(For the next steps, be sure to save changes before returning to the top level: -button.)

Use buttons to:

<u>action</u>	<u>button</u>	
phase		drag up/down on "0" and "1" in toolbar
set the reference		click reference peak
peak pick		box the peaks to pick them
integrate		drag over regions to integrate

10. *Printing:* Enter the printing/layout editor by typing PLOT. Use buttons to position the spectrum, expand it, create insets, etc. When it looks good, click the Printer button.
11. Replace the standard and verify that it has locked.
(If you were using a solvent other than CDCl₃, this will require typing LOCK CDCL3.)
13. Close Topspin and log off Windows. Do NOT turn off the computer.