

NMR Training
Bruker AV-300/400 for novice users

Safety first! *The NMR uses a strong superconducting magnetic field so be sure to remove any metallic objects, watches, keys and credit cards from you before stepping into the magnet room CRN113.*

Login on the corresponding spectrometer computer (screen label) under your group username and password. Two main windows will open. The larger is the NMR software interface. A second window will display the solvent lock window (on the right side). On the left there is a **Browser** information area through which you can see your data files. The main screen is used to display your spectral information. Along the top of this window you will see tabs which refer to other files associated with your data. From left to right they read:

<i>Spectrum</i>	data window for spectral data
<i>ProcPars</i>	process parameters for this data
<i>AcquPars</i>	acquisition parameters for this data
<i>Title</i>	title assigned to this data
<i>Pulseprog</i>	pulse program for this experiment
<i>Peaks</i>	peak listing for this data
<i>Integrals</i>	integral listing for this data
<i>Sample</i>	sample information for this data
<i>Structure</i>	structure info
<i>Plot</i>	<i>printing tool interface</i>
<i>Acqu</i>	<i>real-time acquisition window</i>

The command bar along the top contains a variety of icons to allow control of the spectral display as well as entry to various sub-programs simply by clicking your mouse. Each icon has an associated information line which becomes visible as you place the cursor over an individual icon. Most of the icons for routine use are easily recognized. Icons related to experiment setup are the colored icons labeled.

<i>INSERT</i>	insert sample.
<i>EJECT</i>	eject sample.
<i>NEW</i>	create a new experiment file.
<i>1-H</i>	read the parameters for a normal 1d proton experiment
<i>13-C</i>	read the parameters for a normal 1d carbon-13 experiment
<i>LOCK</i>	lock sample (prompts for solvent)
<i>SHIM</i>	starts the shimming program.
<i>TUNE</i>	starts the atma program to tune the probehead.
<i>ACQ</i>	starts data acquisition.

The use of these functions will be described in the experiment portion. At the bottom of the display you will see a command line and a status bar which includes information about acquisition status, lock, temperature, date and time.

Setting up and running your experiment:

1. Press the **EJECT** button in Topspin and wait until you see sample status indicator mode change to “lift”.
2. Clean the bottom of the NMR tube with wipes provided and put it through the white spinner and push it in until it reaches the bottom of the depth gauge.
3. Place the sample in the shim stack. The nitrogen cushion will support the sample. Walk back to the computer and press the **INSERT** button and the sample will drop down slowly into the magnet bore.
4. Click on the **NEW** icon to create a new data file. An information window will appear. You only need to enter a filename and an experiment number. **DO NOT change any other setting in that window.** Filenames should start with your initials or some identifier that will make the filename easy for you to recognize in the future. Click **OK**. The new experiment is now created. A “No Raw data available, No processed data available” message will be displayed.
5. Select the experiment you wish to run, for example **1H** or **13C**. All other experiments could be found under the Experiments or Probe in the top menu.

6. Click the **LOCK** icon to lock the sample and a list of solvents will appear. Click on the solvent you are using then click OK. A lock is achieved when the lock display trace is in the upper portion of the display, “lock finished” will appear in the info bar. **Your sample must contain a deuterated solvent for the lock system to function.**
7. Click on **TUNE**. This starts the tuning program which takes a minute or so to complete. Do not disturb the program function during this time – when the tuning is complete “atma finished“ will appear in the message bar.
8. Click on the **SHIM** icon to start shimming routine. Ignore any effects visible in the lock display. Shimming is complete when you see “topshim finished” in the message bar. There are also options for **SHIMVT** in case of VT experiments or **SHIM1H** in case of running samples in non-deuterated solvent.
9. If necessary, type **NS** and set the value for the number of scans required depending on your sample concentration. Click on the **Acq** icon to begin the experiment and data acquisition.
Note: Default NS for each experiments assumes a millimolar sample concentration (20-50mg, MW<500) For dilute samples NS needs to be increased in power of two’s considering the square root dependance rule. $SNR \sim \sqrt{NS}$. Also for long experiments do not increase NS more than 1K, use parameter TD0 (loop counter) as a multiple of NS, so default value is 1 if you need 4K for example just set TD0 to 4 and NS 1K, this way your data will not be completely lost in case of a computer glitch or extended power outage.
10. When acquisition is complete (check the Acquisition information window), you can process the data by clicking on the **Process** tab and select PROC sample. If the data requires further phase adjustment, you can enter the phasing routine via the **Phase** tab or phase control icon. The vertical cursor selects the peak to use for zero order correction. Click and hold the mouse button on the **0** icon and move the mouse vertically to complete the phase correction. Repeat this procedure while viewing a remote peak and clicking/holding on the **1** icon to perform the first order correction. When complete, click on the save icon to save the corrected spectrum.
12. If the data requires adjustment of the reference signal, click on the calibration icon and use the vertical cursor to set the new peak reference value.
13. Peak picking results may be created or altered by clicking on the peak picking icon. Use the peak selection icons to generate an appropriate peak list. The list is visible in the spectrum window or you can see the list directly by clicking on the **Peaks** tab.
14. Click on the integral icon to enter the integration routine. Click on the region icon and define the new integral regions. You can calibrate the integration by selecting an integral and clicking the right button to use the Calibrate or Normalize modes.
15. Set the display area for plotting and click on the **PLOT** icon. The plot program will open and you can make further adjustments to the image before taking a hard copy or using File => Export to create another image file. When you have either saved your image file or printed a hard copy, use File => Exit to quit the plot routine. **DO NOT SAVE** the layout when asked.

When finished with your session click EJECT remove your sample, leave the spinner on the console then click INSERT to turn off the air lift. Exit the program by clicking File ->Exit.. LOG OFF your user before you leave.

DATASTATIONS and Remote Access

Presently the NMR lab has two Datastations. You can login into any Datastation using the same username password combination as for the 300 or 400 spectrometers. There will be four drives in your portfolio containing direct access to the current year data from 300, 400 and 600 spectrometer as well as previous years data. They can also be accessed from any computer on campus or off campus through the VPN making a connection to the shared Windows drive.

AV300: 139.57.136.32

DATASTATION-I: 139.57.180.20

AV400:139.57.136.33

AV600: 139.57.136.31

DATASTATION-II: 139.57.136.30