Welcome to the NMR Center

The NMR center in chemistry consists of six Bruker NMR instruments

Field/Name	Location	Comments	Operating Software
300nb (narrow bore)	119 Havemeyer	walk-on, routine $_1$H	Topspin 3.5
300wb (wide bore)	208 Havemeyer		*Topspin 1.3*
400L	208 Havemeyer	$_1$H routine	Topspin 3.5
400SL	208 Havemeyer		Topspin 3.5
500	208 Havemeyer		Topspin 3.5
500 Ascend	214 Havemeyer	separate password required	Topspin 3.5

The best NMRs for $_1$H are 300nb, 400L, and 500 Ascend. The best NMRs for $_{13}$C are 300wb, 400SL, and 500. See the NMR web site for details.

Training to become an NMR user:

All new users must have an introductory demonstration by one of the NMR staff located in room 208 Havemeyer. After you have taken the intro. demo, follow this procedure to obtain a password:

1. On the 300WB instrument, sign up and practice. The NMR staff must be here to log you in. Sign-up for times when they are here (9:00AM-5PM).
2. When you have learned everything on the handout, including manual locking/shimming, manual phasing, integration and adjustment of integrals, and peak picking, make arrangements with John Decatur to watch you, and if you pass, give you a password.

This password allows the user access to the Bruker 300NB, 300WB, 400, 400SL, and 500. Access to the 500 Ascend requires further training (see below). This password only operates on the NMR computers and workstations. At the intro demo a different username/password will be issued that will allow the user to reserve NMR time using the web-based reservation system. Thus, you will have two usernames/passwords.

300 Demo - Carbon
This is an *optional* demo that covers the following topics: tuning the probe, parameters to enhance quaternary carbons, decoupling and NOE, and DEPT spectra. A sign-up sheet is usually posted in 208 Havemeyer. Demos are scheduled when more than 3 or 4 have signed.
400 Demo COSY and NOESY
This *optional* demo provides instructions for the COSY and NOESY experiments. A sign-up sheet is usually posted in 208 Havemeyer. Demos are scheduled when more than 3 or 4 have signed.
500 Ascend
People who desire access to the 500 must first attend the 400 Demo and reproduce the results of that demo.
Variable Temperature, T1, kinetics, HSQC, etc…
These demos are given on an as-needed basis. See John Decatur for details.

1D Proton NMR on Bruker NMRs using Topspin

What is included in this Handout?

- Website, Data Server, Web-based time reservation, NMR prediction
- Quick start guide
- Spectrometer locking and shimming

- Understanding data sets
- Parameters and Commands
- Fixing problems
- Data Server
- Using Cyberduck

- Using Mnova
- Obtaining Topspin
- NMR Sign-up Rules

Website, Data Server, NMR time reservation, NMR Prediction, C14 Certification

Website There is an NMR web site accessible from the main chemistry web page: http://www.columbia.edu/cu/chemistry/groups/nmr. It contains training handouts, information about which NMR can do which nucleus and experiments, the sensitivity of the different NMRs, and more.

Data Server There is a server, nmr22.chem.columbia.edu, from which one can easily download NMR data from all NMR spectrometers to PCs located anywhere. The freeware program WinScp on PCs and Fetch or cyberduck on the Mac can be used to download data easily. Details are in Data Server section.

Processing Software We have a campus license for MNova processing software for user's own computers. Details are in Data Server section. Bruker Topspin software is now free and available here: https://www.bruker.com/service/support-upgrades/software-downloads/nmr.html

Time reservation Time on the NMR instruments is reserved via a web-based system: http://nmr01.chem.columbia.edu/. Each instrument has different sign-up rules that are software enforced. Sign-up rules can be found on the last page of this handout and on the website.

Spectral Prediction We have one shared license for MNova's spectral prediction program. The predicted chemical shifts, esp. for ^{13}C, are superior to those predicted by simple programs such as ChemDraw. Details are in Data Server section.

C14 Certification The New York City Fire Code requires at least one person with a Certificate of Fitness (C-14) to be present at all times whenever a laboratory is in operation, including evenings, nights, weekends, and holidays. **No entry to any NMR lab after hours (outside of 9-5 M-F) for those without a C14, unless accompanied!**

Password, Sign-up Rule, and Reporting Policy

All users MUST have their own passwords. You may not share your password for the web reservation system or the NMR computers/workstations with anyone else. Using another person's password on the NMR computers or signing up for time with another person's username/password to circumvent the sign-up rules is forbidden. **All offenses will result in the suspension of ALL NMR accounts**. For the first offense, the suspension is one day, for the second offense and further offenses, the suspension is five days.

Please tell the NMR staff immediately when an instrument is malfunctioning, or a sample is stuck or broken in the magnet. Never leave a stuck or broken sample in the magnet without a note saying instrument is DOWN, DO NOT USE. Please clean up ALL SPILLS. If hazardous, call Columbia Environmental Health and Safety (212) 854-8749 (9am-5pm weekdays)   or   (212) 854-5555 (all other times; 24 hours/7 days)

Magnet Safety

The magnetic fields within about one meter of the magnetic are extremely strong. **Do NOT bring any iron containing materials (tools, gas cylinders, etc..) near the magnet!** Magnetic media such as floppy disks, tapes, and credit cards can be erased if brought close to the magnet. Mechanical watches (those with hands) can be damaged. See the separate handout on safety or https://nmr.chem.columbia.edu/content/safety-nmr-laboratory

Sample Preparation

The tube should be filled with a **deuterated solvent** to a minimum depth of 5.0cm (about 0.60ml**). Volumes less than 600 uL makes shimming the magnet homogeneity difficult.** Greater depths are O.K., except for variable temperature experiments. The amount of sample required for a proton spectrum ranges from less than 1mg/ml to about 20mg/ml (mw=400). Too much sample can result in a loss of resolution or a distorted spectrum. This includes not just the sample of interest, but any proton source such as protonated buffers, residual protonated solvents, and water. About 5mg/ml is sufficient.   The solution should be free from any solid, such as undissolved solute, or dust. Filter the solution, if necessary. Without a deuterated solvent, locking is impossible.

Quick Start Guide

1. When the instrument is free, the login screen will be shown. Log into Linux. Then, right click and select "open terminal". Type **topspin** in the terminal window to start program. Or click Topspin icon.
2. Lock and shim controls are located on buttons on the BSMS display (Bruker Shim Microprocessor System). Type **bsmsdisp** to open this if it did not automatically open. Or right-click on the lock display icon at the bottom and select **BSMS panel**. On the 300wb, if the BSMS panel gets hung, close and re-open it. Click **lift** to eject sample (or type **ej** and **ij** for insert).   ALWAYS SET DEPTH of tube in spinner with depth gauge!! Failure to set depth will break your sample and/or the instrument. Clean spinner with ethanol.
3. If there is no data displayed in the main window, you may first want to open an existing data set. On the left, you will find the data browser. Click on a disk unit, e.g., 300wb, and drag in a data set from under your username.
4. Type **new** to define the data set. The **new** window appears differently depending on the version of Topspin. The version below, left, appears on the 300wb. Set the USER to your login username and DIR to /300wb. The version below, right, is for all other NMRs. The directory path DIR, visible after clicking options, must include both the username and the disk unit, /300, /400L, /400SL, /500 or /500asc, depending on which NMR you are using. It should be of the form /500/data/YOURUSERNAME/nmr. If you get an error upon saving, the username in this path is likely not correct.
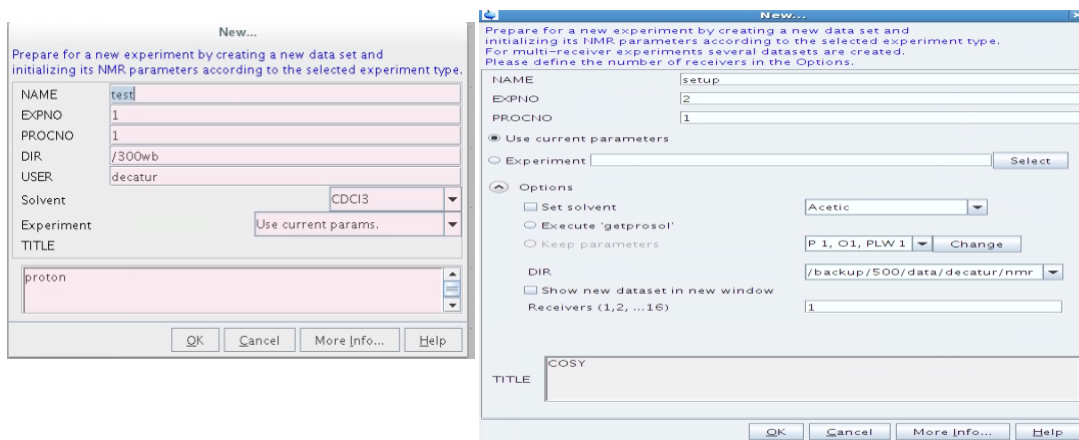
5. Type **lock**, choose correct solvent. Type **lockdisp** to generate lock display or right click on the lock icon ▦ at the bottom and select **locksignal display**. Adjust lock gain such that the horizontal line is approximately two-thirds of the way up the screen.
6. Type **rsh today**. This resets the shims to the standard best values.
7. On 300wb you must maximize lock level using Z and Z2. Type **bsmsdisp** to open the shim display. On all others type **topshim** to automatically shim.
8. Type **rpar protonstd** - reads in default proton parameters.
9. At this point it may be necessary to tune the probe to the NMR frequency. Usually this is not necessary for routine proton. It is necessary for more advanced proton experiments (COSY, NOE) and all X nuclei, such as carbon, experiments. To tune the probe, on all NMRs except the 300wb, type **atma**.
10. Type **start** (macro that includes rga and zg commands) automatically sets receiver gain (rga) and starts acquisition (zg).
11. Type **halt** - stop FID collection before ns scans are completed, or type **tr** - transfer data to processing computer without interrupting acquisition (useful for processing and phasing while acquisition is still in progress).
12. Type **efp**- exponential multiplication (em) and Fouier transformation (ft) and/or phase correction (pk).
13. The following shows the buttons for changing the display:

 Particularly useful buttons include:
This resets the horizontal and vertical display to the full spectrum after an expansion.

14. To phase, Select Process, Adjust Phase. (on 300wb Click phase) Using left button, scroll (keeping it depressed) and to phase. Click save+return .

15. To reference the spectrum, select Process, Calib Axis (on 300wb, Click calibrate). Place cursor on peak and click left button.

16. To integrate, select Process, Integrate. (on the 300wb Click integrate). Click to turn on integral region definition; press left button and drag to define. Right click under integral to select. Adjust selected integral tails to be parallel with baseline by scrolling bias and then slope . A properly defined integral should extend beyond the apparent ends of the peak (if there is no other adjacent peak). A properly phased integral should be horizontal before and after the peak. If peaks are very close together, phasing the integrals separately may not be possible. The best procedure is then to define one large integral that covers the

entire spectrum, adjust bias and slope, and then "chop" it into smaller integrals. To do this "chopping" click .

17. To calibrate, right click under integral and select calibrate. Click save+return .

18. To peak pick, select Process, Pick Peaks. (on 300wb, Click peak peak). To turn on peak region definition, click so it is highlighted. With left button drag the box to include the top of all peaks to be picked. To add a peak, click and select peak with cursor and left click. Click save+return .

19. To print exactly what is on the screen, select File, Print (does not work on 300wb). Select print active window and click OK. Select printer; for room 208 select room208_letter or room208_11x17. In room 119, substitute 119 for 208 in above. Paper size MUST match letter (8.5x11) or engineering (11x17). Click print.

20. To print via Topspin Editor, Select "File, Print with layout – start Plot Editor (plot)". Choose appropriate LAYOUT (see printing section). Click OK.

21. To read in existing data, e.g. on datastation computers, drag data from the browser window into the main window. If your directory is not listed, right click and select "Add New Data Dir…" and add either /300nb, /300wb, /400L, /400SL, /500 or /500asc .

22. When finished put standard sample (any sample will do) back in magnet. ALWAYS SET DEPTH with depth gauge!! Failure to set depth will break your sample and/or the instrument.

23. **Exit the Topspin program (and wait for it to completely quit) BEFORE logging off the computer.** To log off Linux, click in the upper right corner, then click on your username, and then select logout. It is very important to exit the program in the correct manner. (Logging off the computer first leaves processes running and causes problems.) To exit Topspin, type **exit**, and then click on *OK*. To log out of Linux, one clicks on the upper right corner power icon (See adjacent image.) But logout is not immediately displayed. One MUST click on the username, which THEN displays the Log Out option.

24. Questions? READ the rest of this guide BEFORE ASKING
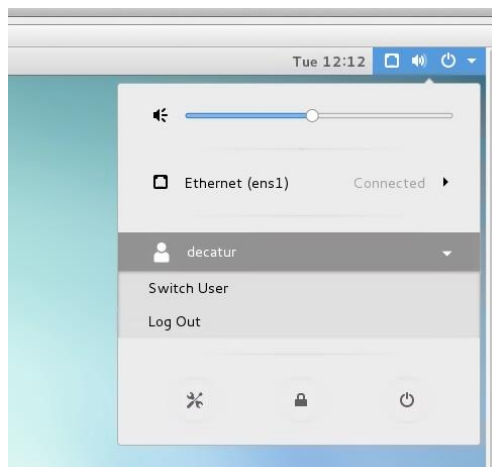
Shimming

If necessary, reset the shims to the standard best values, by typing **rsh today**. Type **topshim** to automatically shim. Or type **topshim 1dfast** for a faster, less extensive shimming. **Topshim convcomp** helps if the sample is not yet at thermal equilibrium and topshim fails. **Topshim shigemi** works for Shigemi tubes. The following manual procedure is sometimes required:

Optimize Z and Z^2. The goal is to maximize the lock level by adjusting the shim values (lock gain is not a shim value; use lock gain to keep the lock display in the middle of the display range). Adjust the shim values slowly since there is a delay in the lock response.

1. Adjust Z1 for maximum lock level.

2. Maximize Z2,
3. Repeat until no further improvement is seen. Reduce lock gain if the lock signal goes to the top of the screen. Always end with Z1.
4. Shim X, Y, XZ, and YZ if necessary. (you must not spin the sample)

Do not adjust Z3 or Z4. The proper value of Z4 requires hours of shimming. If you have followed the above procedure, any change in Z4 should reduce the lock level.

Data Sets

All data is automatically written to disk. If you do not change the data set name before acquiring data, the new data will over-write existing data. Thus defining a data set is one of the first steps and should be done prior to reading in parameters. Type **new** to define a new data set.

These parameters must follow these guidelines:
♦ *NAME* - name of data set - usually describing your sample. *The use of several special symbols in the name, such as /, !, $, * and . are not permitted and will cause errors*.
♦ *EXPNO* - experiment number (can only be numbers)
♦ *PROCNO*- process number (can only be numbers)
♦ *DIR* – directory where your data is to be stored. Included in the path should be YOUR username and the data disk. An example is /500/data/YOURUSERNAME/nmr. If you get an error upon saving, the username in this path is likely not correct.

Each instrument has a different disk unit and their names are given in the following table:

	NMR Instrument			
	300nb (rm 119)	300wb (rm208)	400L or 400SL	500 or 500 Asc
Disk Name	/300	/300wb	/400L or /400SL	/500 or /500asc

The parameters defining the current data set (Name Expno Procno Dir) are found at the top of the Topspin data window. The parameters represent directory names on the computer's disk and are arranged in a tree structure with the disk unit at the top. For example, under a user name (yours) there may be many names of data sets. Under one name, there may be many experiment numbers, and under a given experiment number, there may be many process numbers.

Raw data, that is, FIDs, and parameters are identified at the experiment number level. Completely independent data can be identified by different names or experiment numbers. Spectra are identified at the process number level. Different process numbers are only used for different processing (window functions, FT, difference spectra) of the same raw FID.

Parameters

Acquisition and processing parameters can be accessed from the AcquPars and ProcPars tabs in the data window, respectively. Alternatively, each parameter may be changed by simply typing its name, and then changing the value. Some important parameters are as follows:

- sw - spectral width
- o1p - transmitter center (center of spectrum)
- ns - number of scans
- rg - receiver gain
- d1 - relaxation delay
- p1 - pulse width (for simple 1D acquisition)
- aq - FID acquisition time
- td - number of points used to define FID

- si - number of points used during FT (can be different from td)

Processing Commands

The following commands are useful for 1D NMR:
- **em** - exponential multiplication on the FID, uses the parameter LB. This improves signal to noise at the expense of resolution.
- **lb** - this controls the degree of broadening added and affects your signal-to-noise. To see its effect, simply change its value and re-Fourier Transform with **ef**.
- **ft** - fouier transform
- **ef** - combines **em** and **ft**
- **pk** - phase correct, applies the last phase correction to the spectrum. Useful when you have phased a primlimary spectrum, (with only a few scans) and wish to apply the same phase correction to the final spectrum.
- **efp** - combines **em, ft**, and **pk.**
- **absn**- automatic baseline correction without integration
- **apk** – automatic phase correction

Multi-spectra Mode

Click [icon] and drag spectra into the data window. There is no limit to the number of spectra that can be displayed. To space them uniformly, click [icon]. To give them a horizontal offset, scroll [icon]. One can print directly from this window using the Ctrl P print method but one *cannot* start the Plot Editor from multispectra mode and have more than one spectrum displayed.

Plotting using Topspin Plot Editor

To print using the versatile plot editor, select File, Print. Select "Print with layout – start Plot Editor (plot)". Sometimes the plot window appears blank at this point. Simply click on another tab, such as FID, and then click on plot tab again and your spectrum will appear. You can print on either 11x17 or 8.5x11 with this method. The paper size, printer location, and whether the data is 1D or 2D depends on the LAYOUT chosen. Choose a LAYOUT from the table below.   Always make sure paper size corresponding with layout! Click OK.

Valid LAYOUT files for Plot Editor	
1D119_11x17	1D119_8.5x11
2Dhomo119_11x17	2Dhomo119_8.5x11
1D208_11x17	1D208_8.5x11
2Dhomo208_11x17	2Dhomo208_8.5x11
2Dinv208_11x17	2Dinv208_8.5x11

On the 300wb, the plot editor is a stand-alone program. On all other NMRs, it is part of Topspin.

Data Stations, off-line processing

To increase the throughput of the spectrometers, use the data station for processing whenever someone is waiting for the instrument. Do not sign up for NMR time if you are only processing or plotting data. When using the data stations, your data is not "transferred" or copied to the other computer but is only accessed over the network. Topspin processing software is now FREE and available from Bruker for download.

Problems

The following are common problems on Bruker spectrometers and their solutions:
1.The commands topshim, rga, start, or zg do not respond or are "hung". The solution: Type ii. If this fails, exit Topspin, restart topspin and type ii again.
2. If the above fails, open the console door and with a pen, push the IPSO reset button. Wait one minute and type ii in Topspin. Type ii again if there are errors.

Archiving Data – Deletion Policy

Data is currently not being deleted but this is subject to change. Data may be backed up using CD, DVD, USB memory sticks, or downloaded to PC from the NMR data server. See NMR data server handout for details.

NMR Processing on PCs outside the NMR laboratory

A server stores the NMR data from all spectrometers and can be accessed from PCs anywhere. For processing, we have a campus license for 200 users for MNova software which runs on Mac, Windows, and Linux. Bruker Topspin software is also free and available from bruker.com

DATA SERVER: Downloading Data from the NMR server

To download your data (and MNova license) from the NMR server onto your mac or PC, you need a client scp program. On Windows, the free SCP client program is WinSCP or Cyberduck. On the mac, it is Cyberduck.

WINDOWS

Follow instructions below for Cyberduck or for Winscp follow these below

Download WinSCP from https://winscp.net/eng/download.php. Choose the latest version and the "installation package" type of download. Launch WinSCP and enter the following: hostname is nmr22.chem.columbia.edu (or 128.59.112.134), port is 22, enter your username and password that works for the NMR computers, and select SCP type of transfer. Leave blank the space for private key file. Click login. Navigate to reach your desired source and destination directories. For the server, navigate to the root directory (/), and then choose the disk you want: 300, 300wb, 400L, 400SL 500, or 500asc and then your name, and then data set. To download, press F5, or right-click the mouse and select copy (F5).

MAC

Download and install Cyberduck from http://cyberduck.ch/
After it downloads, click on cyberduck.zip to decompress it and then move it to your application folder. After Cyberduck is launched, Choose Open Connection from the top menu window and the following window opens:

John Decatur   VERSION 3.0   1/10/2020
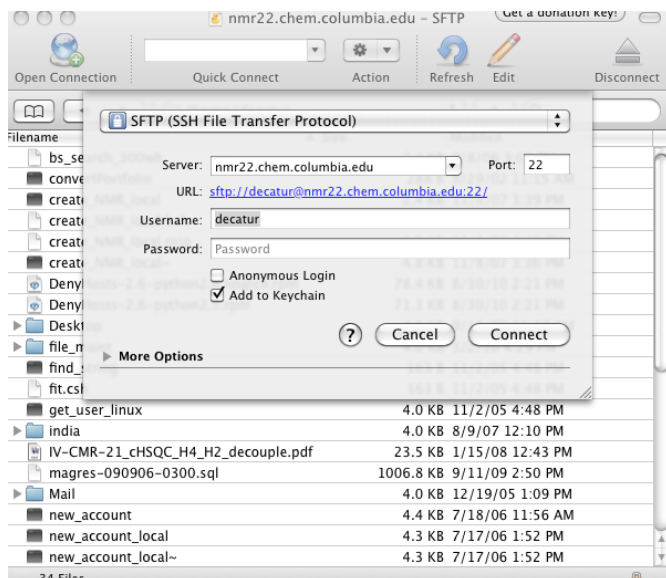
At the top of this menu, chose SFTP (SSH File Transfer Protocol) and enter for Server: nmr22.chem.columbia.edu (or 128.59.112.134), Port: 22 and your username/password on the NMR computer. Leave SSH Private Key: None. Click Connect.

The first time you log in, you will receive the following Unknown fingerprint message. Click Always and Allow.
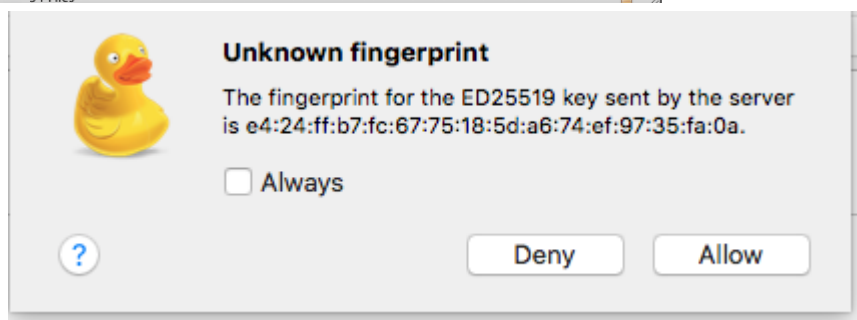
If successful, you are logged in and a (home) directory and its contents are displayed. Using the navigation bar (as shown below in circled red),
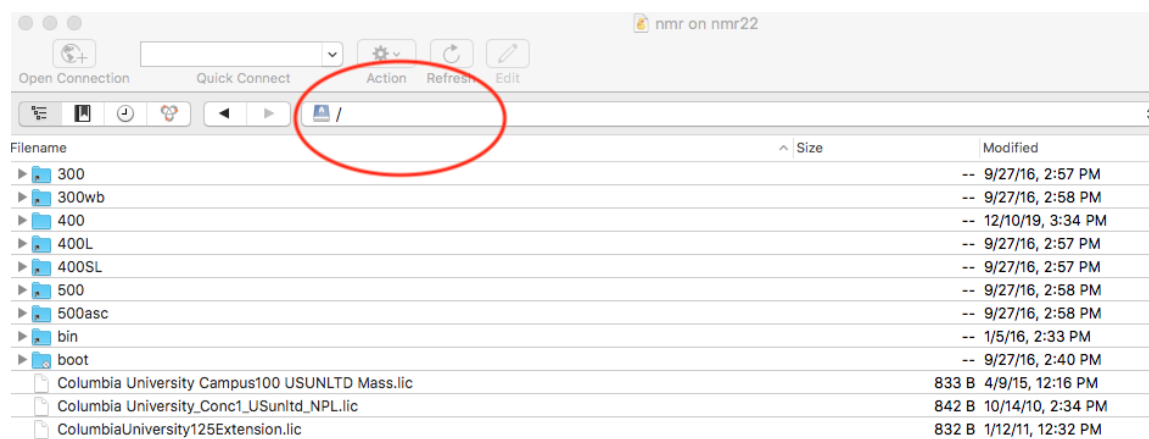
navigate to the data directory /. In this directory, there are folders for all the different NMR as well as the MNova license files. Download all the license files (they have extension .lic).

To download the license file, select the file and either right-click with mouse or click the action icon, and select Download to…. I recommend a save location different from Downloads.

To download your NMR data, navigate to , for example,/400 directory and then your username directory, such as /400/decatur. Select the data you want from this directory and download. Do not enter sub-directories and then download because all the parameter files are needed.

One can bookmark all the information entered into Cyberduck so you don't need to repeat it next time. While still connected, from the main menu Bookmark, select New Bookmark and accept or change the information.

The NMR server is completely updated every night between 1 AM and 5 AM. Thus, data collected today will be always available tomorrow. If you need access to recently acquired data you can manually update the server for only your data. On one of the NMR linux computers, open a terminal, or shell, and type update and follow the directions. You will be prompted for your password, which is the same as on the NMR computers. The update may take a few seconds or minutes depending on how much data you have. Do not logout of the computer until it is finished. Only one disk (400L, 300wb, etc..) is updated at a time. From a spectrometer computer you may only update data from that particular spectrometer.

Only NMR data directories are transferred to the server, not your home directories. If you wish to have access to pdf or other graphic files created by Topspin, then you must store them in the corresponding data directory, and not in your home directory.

Obtaining MNova and Installing License

Columbia has a campus license for MNova NMR processing software, which runs on Mac, Windows, and Linux. The following assumes you have already downloaded the license.

1. You can download the MNova software from http://mestrelab.com/download/mnova/ and install.
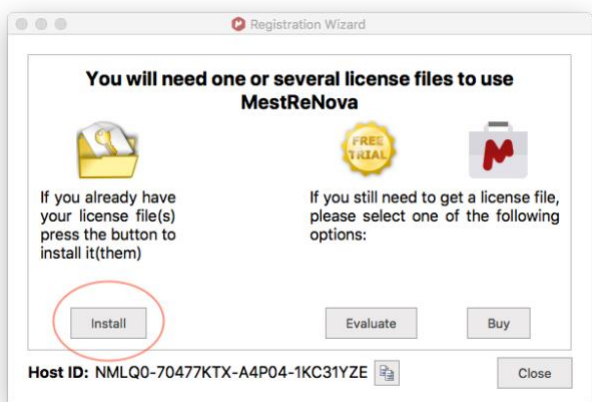
2. Launch MNova and the Registration Wizard auto opens:

Click install and find your license where you saved it in the steps above and click Open. You should get a successful message. ***To activate the license, you MUST exist MNova and restart.*** If you need to return to the registration wizard, find it under Help, Get/Install licenses…..

All 3 licenses should be installed and they need to be installed one at a time.

When MNova now starts, it will ask if you want the modern interface. Always choose the modern interface.

Using MNova

Opening Data

The easiest method is to drag a file directly into MNova. You may also use File, Open from the top menu.   If you like Topspin's Data Browser, then one can enable a very similar feature by checking the Data Browser box under View.

Your data is automatically Fourier Transformed and phased.

Before You Begin

Cursor Mode: FYI, In MNova, the cursor shifts into different modes when you click on, for example, the expand icon, . The cursor then creates expansions. This continues until you hit Esc.

Pages: From the View menu, check the box Pages to show the different open spectra on the left side, like PowerPoint.

Page Setup for Printing: From the File menu, select Page Setup and change the Page Size to Letter and Orientation to Landscape.

Manipulating the View

Use the icons on the View tab to manipulate your spectrum and display:
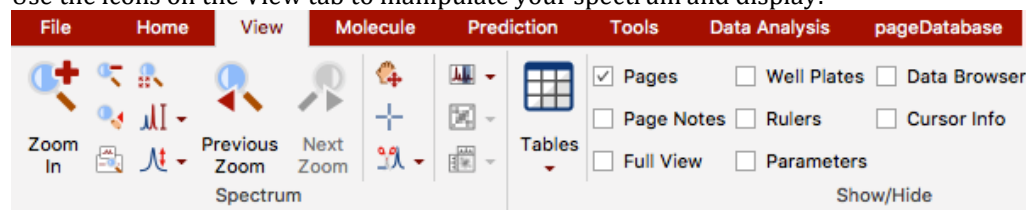
To zoom or expand: or type z once for horizontal expansion, twice for vertical expansion. Esc to return to normal mode.

To go to full spectrum: or double click the spectrum when cursor is in "normal" mode.

To change vertical intensity: The mouse wheel controls vertical display. Or

To set (or reset) the vertical display:

To create an expansion region inserted onto the spectrum:

To list the parameters: check the box Parameters. This opens a separate window of parameters. Click

Customize tab, if desired. To write these parameters to the spectrum, click Report,

To cut, or remove, parts of an empty baseline: . This is scissor mode and the cursor then defines areas of the spectrum to be out of view. Type V (shortcut) to switch to restore mode.

Processing

Click the Processing tab under NMR to show processing steps
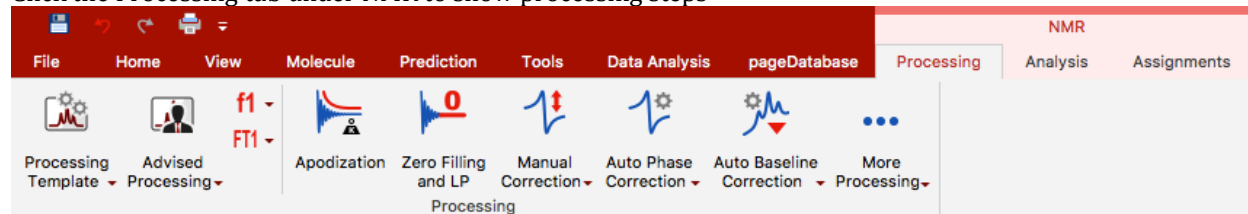

By default, Apodization, Fourier Transformation, and Phase Correction are automatically performed.

Many icons have two parts with the lower part giving you options. For example, Auto Baseline Correction, , has two downward arrows. The bottom downward arrow brings up a menu of baseline correction options while the upper main part immediately executes the default auto baseline correction.

Manual Phase Correction:

Manual correction may be necessary, especially for $_{19}$F spectra. To manually phase, click and place the cursor into the purple colored box, and mouse down and hold down with left button for zero order phasing. Zero-order phasing affects all peaks the same. Only focus on the largest, highlighted peak. Mouse down and hold down with right button to adjust first-order phasing. 1$_{st}$-order phasing applies a linear correction with origin at the largest peak.
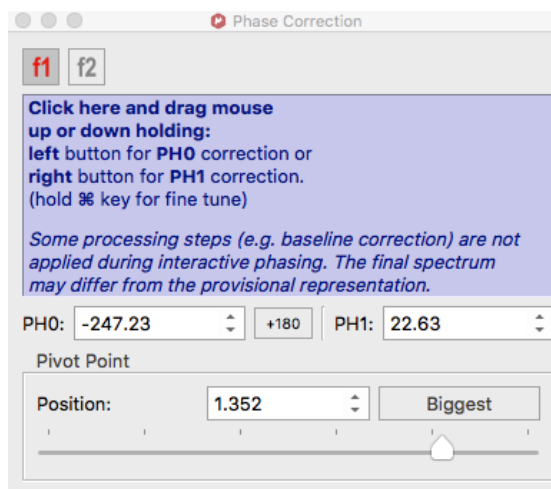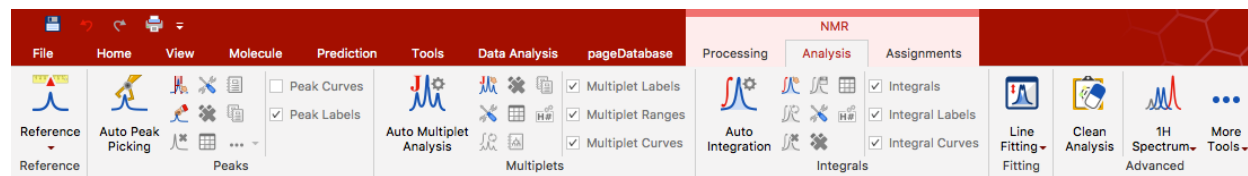
Baseline Correction:

To obtain accurate integrals, baseline correction is necessary. By default, MNova does not perform automatic baseline correction. Click the top part of to immediately perform auto baseline correction.

Analysis

Click the Analysis tab under NMR to show analysis possibilities.

Referencing:

A good idea is to expand around the peak to be referenced. Then click Reference, , and place your cursor on the peak of interest which will be highlighted. Double click and you may enter the new shift. An alternative, useful option is to select Reference by Solvent and then a peak that matches common solvents is used to reference the spectrum.

Peak Picking:

Click Auto Peak Picking for fully automated peak picking. A better option is to use manual threshold, , which allows one to set different thresholds for different groups of peaks. The curser draws out a box that includes the top of all peaks to be picked. Peak-by-peak mode, , allows one to add additional peaks directly by selecting them.

Integration:

Click Auto Integration, ![icon] for fully automated integration. A better option is to use manual mode, ![icon]. In manual mode, the cursor is used to define the beginning and end of the integral. The first integral is always normalized to one but one can edit the normalization by either double-clicking the integral or right-clicking the mouse while the cursor is on the integral and select edit. Entering a new normalization value for one integral will recalculate all integrals. One can also adjust the bias and slope of the integral but if the baseline is flat, this is unnecessary.

Multiplet Analysis:

Use manual mode, ![icon], to choose multiplets one by one. Mulitplets are analyzed by first-order analysis (doublet, doublet of doublets, etc…). Double-clicking on the multiplet analysis box, opens the Multiplet Manager that lists the results. One can list onto the spectrum the full multiplet analysis in a journal-ready suitable format by clicking Report Multiplets, ![icon].

Annotating your Spectrum:

Under Edit, Annotate in the main menu, select Text. Click where you want to add text and start typing. This text box is attached to peaks so when you move the peak intensity, the text may go off the page.

To create a text box that is stationary, you must copy/past a text box from a different page. To create a new page, under the main menu select Edit, Create New Page. Click inside the new page and then create a text box in the new page and then copy/paste the text box.

Printing and Saving

Saving:
In the main menu, select under File, Save As…. Unlike Topspin, you need to save your work in MNova or it will be lost upon exit. You can save in many formats, as shown below.
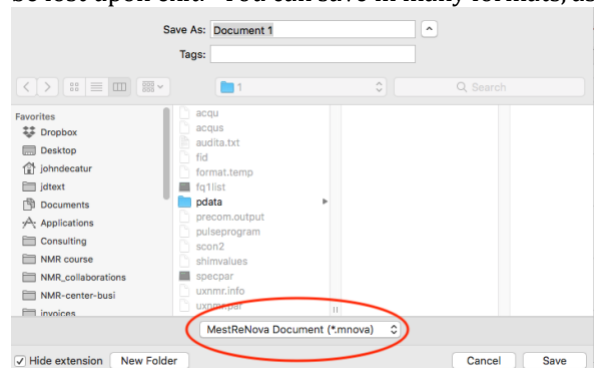
The default is the MestReNova Document format that stores all your current work in MNova. For basic printing, you can save as a pdf. To include in PowerPoint or Word, a JPEG is preferred.

Printing:

Select File, Print in the main menu. Prior to printing, make sure the pager size is set correctly to letter within Page Setup…

Quick start guides for NMR processing using MNova are here:
http://resources.mestrelab.com/category/starting-guides/
http://resources.mestrelab.com/category/resources-by-product/resources-nmr/

Spectral Prediction with MNova

MNova includes a plug-in for Modgraph's prediction software. Modgraph in turn uses several academic programs for its prediction and is superior to ACD labs or Chemdraw. See http://www.modgraph.co.uk/ for a full overview. For C13 shift prediction, the traditional reference database approach is combined with neural networks to give accurate values. Stereochemistry is explicitly considered. For 1H shift prediction, either the traditional approach (called increments) or a conformer approach (CHARGE program) is an option. CHARGE considers partial atomic charges and steric interactions. To read about CHARGE, see www.modgraph.co.uk/product_nmr_proton.htm.

1. Using a drawing program, draw a structure. To show stereochemistry, simply use the UP/DOWN bonds in the structure. (ChemDraw seems to work fine but in the past only versions pre ChemDraw 10 worked. You may have to save as file type MDL .mol file.)
2. Start MNova.
3. Bring a structure into MNova. Files can be drag-and-dropped or brought in via the open dialogue, or copy and pasted.
4. Highlight the structure by clicking on it. Under Molecule, select either Predict 1H spectrum or 13C spectrum. The calculated spectrum will be displayed, which can be treated as an ordinary spectrum (can be integrated, etc…)
5. When you pass the cursor over the atoms of the molecule, the corresponding peaks in the spectrum are highlighted in blue.
6. To display the assigned calculated peak list, highlight the spectrum and select View, Tables. At the bottom of the table, select the 13C Prediction or 1H prediction list. To create the list as an object on

   your spectrum, click the report icon,

Configuration Information for MNova Prediction

Under Prediction, Prediction Options, make sure Predictor is set to Modgraph NMRPredict Desktop. Click on Predictor Properties:
The default parameter values are fine but can be changed, if desired. For 1H, algorithm can be either increment or (CHARGE)conformer, or best, which is a combination.

Obtaining Topspin

Topspin works on Mac, Windows, and Linux and is free for academics! You download directly from Bruker's website.

Go to bruker.com. You must register first. Click login and at the bottom select Register for an account. Once you receive your account, login and return to the bruker.com main page. Then, click Service, and under Support & Upgrades select Software Downloads, as shown below:
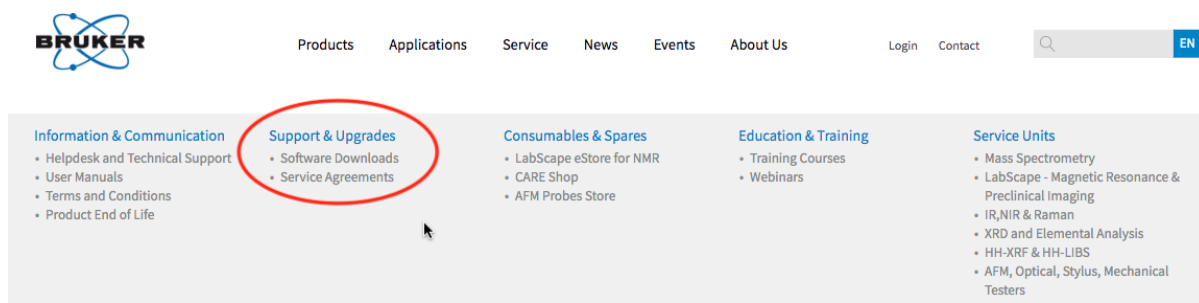
Then, select Nuclear Magnetic Resonance Software Downloads and the page below opens. From this page, you can access the software downloads and the license code. First, obtain the License code.

Click on Free Academia license and then Request personal license ticket now.   The 25 character code is generated immediately and is displayed on the screen. Copy it to the clipboard and write it on paper

Second, you need to download the appropriate software and install.   Return to the NMR Software & Downloads page. On the left side, select either Topspin for Windows, Linux, and MacOS as appropriate.

A page titled Topspin Processing Only Package opens. Select the highest numbered version, such as Topspin4.0.7 Processing Only. Download and install the software. The installation will ask you to enter the NMR administration password. This step *creates* the password for the program's admin functions. I recommend using the password *topspin*. Save this password for future use. Once you start Topspin, select "Activate this installation now" and follow instructions: you will need to use the license code given before.

Sign-Up Rules for NMR Instruments

Rules for Bruker 300nb

	Maximum usage period	Sign-up period
8am -10am	2 hours	36 hours in advance
10am-8pm	open use, maximum 2 samples or 10 minutes	no sign-up
8pm-midnight	2 hours	36 hours in advance
midnight - 8am	no limit	36 hours in advance

** During open period, spectra should be processed at the data stations.

Rules for Bruker 300WB and 400SL and 500 (in room 208)

	Maximum usage period	Sign-up period
MONDAY-FRIDAY		
8am - 5pm	3 hours	36 hours in advance
5pm - 10pm	5 hours	36 hours in advance
10pm -8am	No limit	36 hours in advance
SATURDAY - SUNDAY (except for solid-state work)		
9am - 10pm	6 hours	36 hours in advance
10pm - 9am	No limit	36 hours in advance

Rules for Bruker 400

	Maximum usage period	Sign-up period
8am -10am*	2 hours	36 hours in advance
10am - 8pm	2 ten min. slots**	2 hours before
8pm -12 midnight*	2 hours	36 hours in advance
12 midnight - 8am*	No limit	36 hours in advance

**Total maximum is 2 hours per day for this period.

All exceptions must be approved by John Decatur