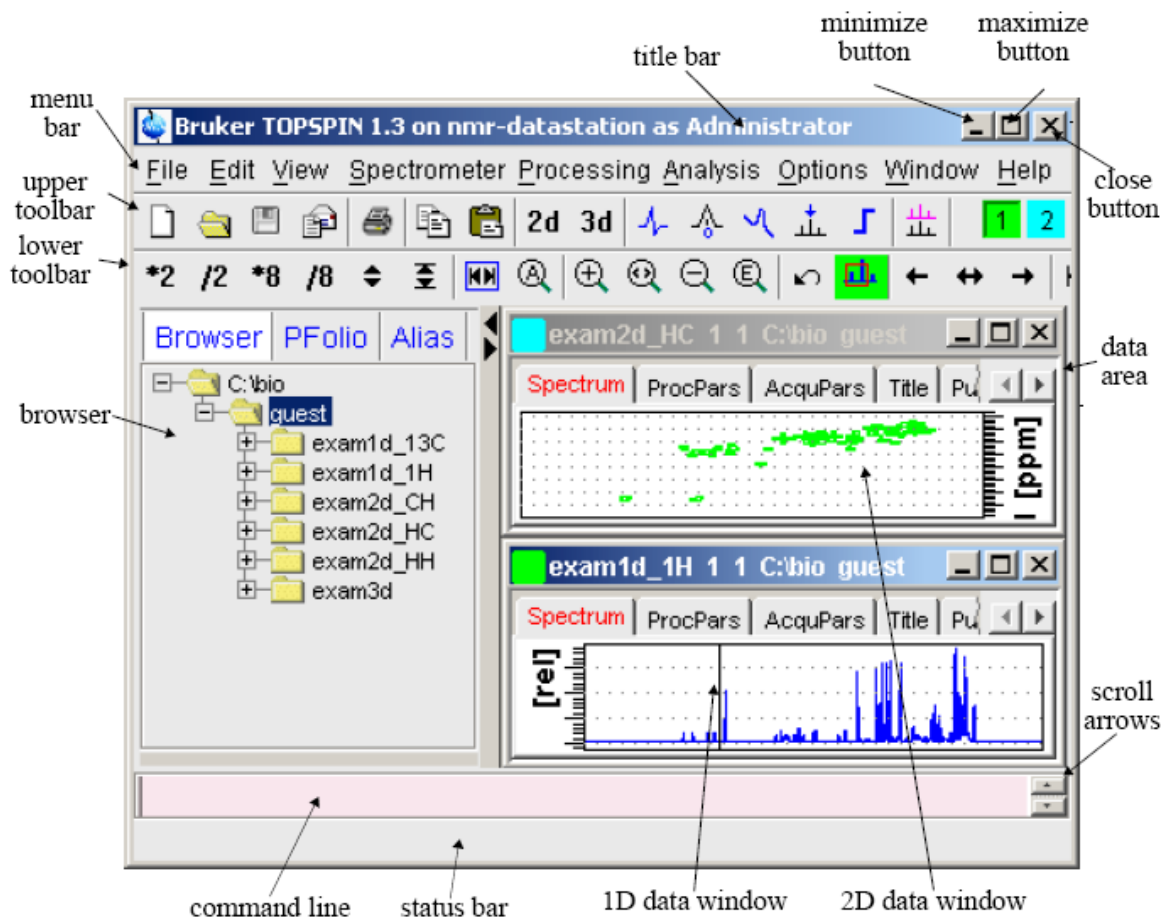


# 1D Solution NMR Procedure

(Bruker AVANCE Machines running TopSpin under WINDOWS XP)


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## \*\*\*Safety Issues\*\*\*

 If you have metal implants, DO NOT do NMR yourself;

 Take everything ferromagnetic or vulnerable to magnetic field, such as mechanic watches, cellular phones, keys, credit cards, bank cards, tapes, computer disks, etc., out of your pockets and put them somewhere away from magnets;

## Table of Contents

<b>1D Solution NMR Procedure.....</b>	<b>1</b>
<i>I. Sample preparation.....</i>	<i>3</i>
<i>II. Sign onto Logsheet.....</i>	<i>3</i>
<i>III. Start TopSpin Software.....</i>	<i>3</i>
<i>IV. How to Load/Change Samples.....</i>	<i>4</i>
<i>V. <sup>1</sup>H-NMR Setup and Data Acquisition.....</i>	<i>5</i>
<i>VI. NMR Data Processing.....</i>	<i>12</i>
<i>VII. Finishing up.....</i>	<i>16</i>
<i>VIII. Acquiring and Processing a 1D <sup>13</sup>C Spectrum (<sup>1</sup>H Decoupled).....</i>	<i>18</i>
<i>IX. Finishing up.....</i>	<i>19</i>
<i>X. Appendices.....</i>	<i>20</i>
A. BSMS Panel (Keyboard).....	20
B. Lock details and troubleshooting.....	20
C. Shimming Magnetic Field --- <b>VERY IMPORTANT</b> .....	21
D. 1D Data Processing Details.....	22
E. LB and Window Function.....	25
F. Multi-Display.....	25
G. Plot Editor.....	25
H. Spin Dynamics and Relaxation.....	27
I. Probe Tuning and Matching.....	28
J. Online NMR Book and Bruker NMR Encyclopedia.....	30
K. Requirements for Access to the MRL NMR at CNSI.....	30

## I. Sample preparation

1. NMR tube of **5mm** or **10mm** in OD (typically 7" long) are used and available from:

- i. Chemistry Department Stockroom, UCSB (Phone: x2107)
- ii. Aldrich (Phone: 800-558-9160)
- iii. Wilmad (Phone: 800-220-5171).



5mm NMR tubes are much easier to shim and give better resolution, while 10mm ones are used for more sample volume and better sensitivity. Most of the time, 5mm tubes are used.

2. Samples are dissolved in **deuterated** solvents (~0.1 mM in concentration, and at least 0.5 ml and 4 ml in volume for 5 mm and 10 mm tubes, respectively, or 4 cm in height) for three purposes. Deuteration

- i. removes solvent  $^1\text{H}$  signals which would otherwise dominate the  $^1\text{H}$  spectrum.
- ii. provides a lock signal.



Lock is a deuterium NMR process that the spectrometer uses to prevent the magnetic field from changing during the course of nmr experiments, thus locking the spectrometer.

- iii. provides an internal reference for the spectra of  $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{29}\text{Si}$ ,  $^{31}\text{P}$ , etc., rendering addition of reference standards such as TMS unnecessary.


## II. Sign onto Logsheet


### Enter

1. your name
2. your advisor's name and department
3. your recharge account number (in the format: 8-4xxxxx-xxxxx-3)
4. your start time
5. **(Do this at the end of experiment:** your stop time and duration of experiment)
6. **(Do this at the end of experiment:** Status of instrument and report problems if any)

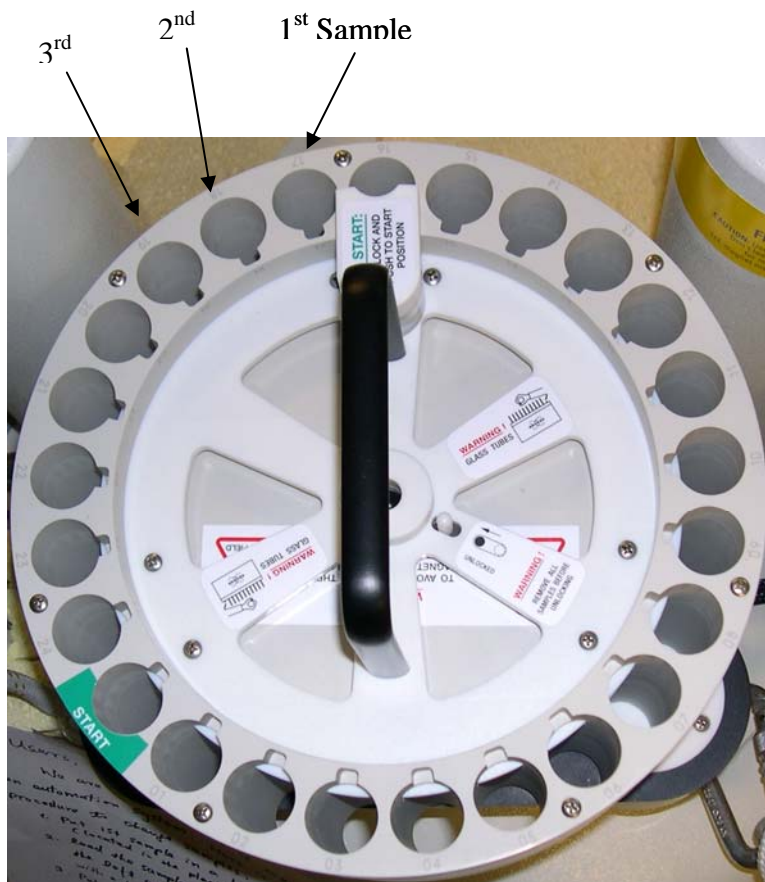
## III. Start TopSpin Software

1. Make sure that the spectrometer is idle by looking at the computer. If yes, proceed to Step 2 below (if no, either wait, talk to the user, or do something else)
2. Login into the WINDOWS computer  
Type your username, hit the Tab key  
Type your password, hit return

3. Double click the TopSpin icon  on the desktop, the last dataset from your previous login session will appear. (The first time you login, you may see a license agreement window. Choose *Accept* and then *OK*).

 Power of right-click provides more functions and options. If you cannot find something you want, try the right mouse button.

#### IV. How to Load/Change Samples.



The 24-slot sample tray

1. Load samples onto the sample changer as below:
  - i. Put samples in the blue spinners, measure depth with the depth gauge, and clean the bottom half of sample tube with napkin while holding the top half;
  - ii. Load them in the slots sequentially to the left of the white plastic piece with words "**TO START:**", starting normally with the first slot. If necessary, empty slots can be left among samples of different batches or users.
  - iii. Please check that all samples have been correctly loaded and record the position of samples (necessary only for automation).



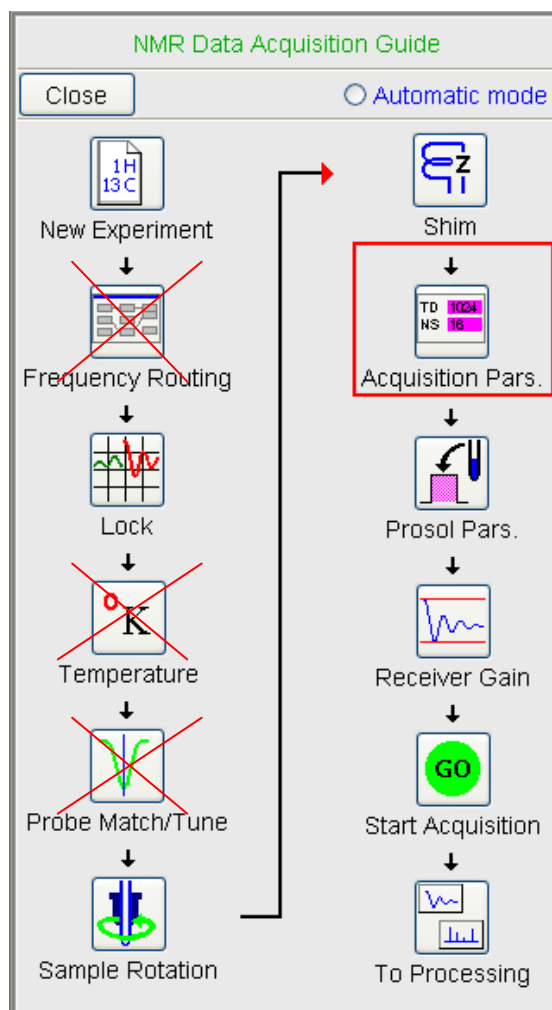
The rotation of sample tray is controlled by air either manually or automatically, depending on whichever mode is set and used. The rotation is clockwise when viewed from the top.

2. Push “Lift ON/OFF” button on the BSMS panel (see [Appendix A. BSMS Panel \(Keyboard\)](#)). The D<sub>2</sub>O sample will come up and the sample tray will rotate. The 1<sup>st</sup> sample will be at the position to load and descend to the top of shim stack (the usual position without sample changer);
3. Press “Lift ON/OFF” button on the BSMS panel again to let the sample go down into the magnet. Wait for 10~15s for sample status LED to show DOWN (green);
4. Place the D<sub>2</sub>O sample in the next available slot following your sample(s), which would be the 1<sup>st</sup> slot if you have only one sample to run;
5. Run experiments using the procedure below for the 1<sup>st</sup> sample;
6. Once done with the 1<sup>st</sup> sample, repeat steps 2, 3, and 5 for the next sample and other samples if available;
7. Once done with all samples, do steps 2 and 3 to load the D<sub>2</sub>O sample, and lock the magnet for wrap-up as described in [Finishing up](#) at the end of the procedure.


## V. <sup>1</sup>H-NMR Setup and Data Acquisition


Once a sample is loaded into the magnet, an <sup>1</sup>H-NMR experiment can be performed as described below.

Click *Spectrometer* → *Data Acquisition Guide*, the flow chart below appears at the right side of the data area:



By sequential clicks on all the buttons except **Frequency Routing**, **Temperature**, and **Probe Match/Tune**\*, the Acquisition guide will walk you through the data acquisition process interactively. At each step, a dialog window will open to offer you the available choices and required parameters.

\*  For simple routine <sup>1</sup>H-NMR experiments, those three steps are not required and therefore will be skipped.

1. Click on **New Experiment**  (or *File* → *New*) [*New*] (Words in brackets are the corresponding commands): a window pops up where you can


Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type.


NAME	1H Cyclosporin
EXPNO	1
PROCNO	1
DIR	C:\Bruker\TOPSPIN
USER	asmith
Solvent	CDCI3
Experiment	Use current params.
TITLE	1H zg Cyclosporin CDCI3


OK Cancel More Info... Help




set:

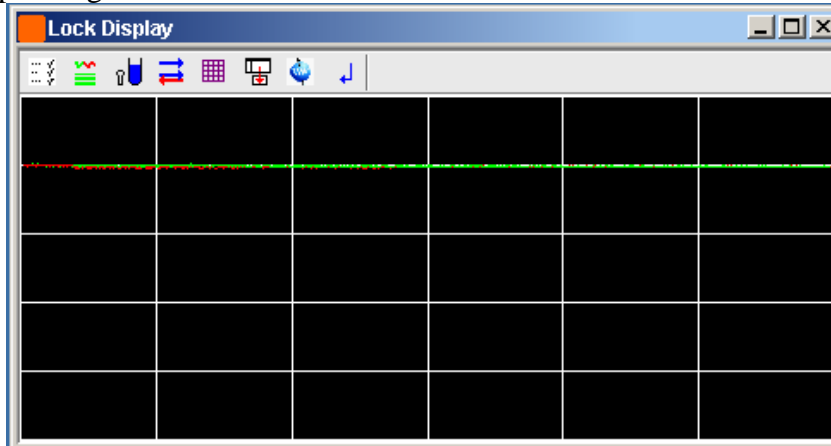
Name*:	e.g. <sup>1</sup> H_AlaGlyBCP	(meaningful or descriptive)
EXPNO*:	1	(start with 1)
PROCNO:	1	(start with 1)
DU:	C:\Bruker\TOPSPIN	(don't change)
USER:	(your loginname)	(e.g. smith)
TYPE:	nmr	(don't change)
Solvent:	e.g. CDCI3	(solvent your sample uses)
Experiment:	e.g. PROTON	(experiment you want to do)
		or choose "Use Current Params" if you want to run the same experiment as the current data in display <sup>\$</sup> .
Title:	(any information useful for current sample, project, and/or experiment)	

: To display an old dataset, go the browser on the left, find and right-click on the desired dataset, and choose "Display" or "Display in a New Window".

\* : IF YOU DO NOT CHANGE EITHER THE NAME OR THE EXPNO OF YOUR DATASET, YOU MAY OVERWRITE YOUR OLD DATA AND LOSE IT FOREVER.

- Click on **Lock** , which opens a solvents table. Select the solvent for your sample followed by **OK**. The locking process will start automatically and several lock parameters are adjusted. Several LEDs on BSMS panel (See [Appendix A: BSMS Panel \(Keyboard\)](#)) will flash and then go off. Finally the lock LED stays on after flashing for ~10s. In the lock window below, a flat line with noise sweeps back and forth in alternative red/green colors. [See [Appendix B: Lock details and troubleshooting](#)].


- Click on the middle button of icon    in the upper toolbar to open the lock display window. If it is not in the front, bring it up by clicking on the corresponding icon at the bottom of screen.

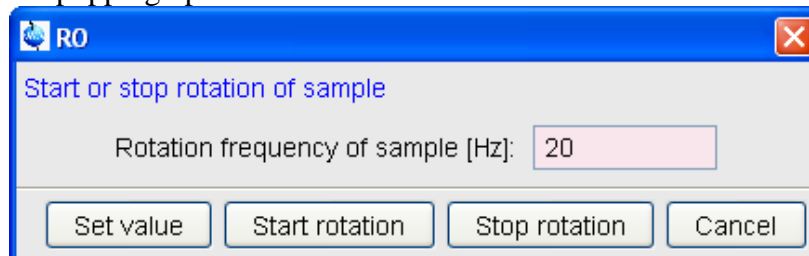


- (Do this only if sample rotation is not on, i.e. the “Spin ON/OFF” LED on the BSMS panel is not lit)




: Iconize or relocate the lock display window if you can NOT see the flow chart)

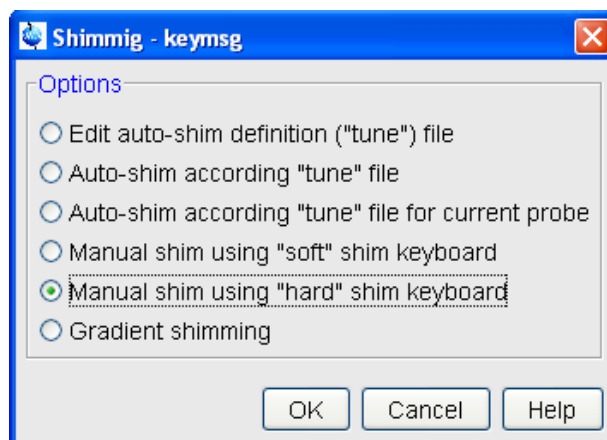
Click on **Sample Rotation**  and set “Rotation frequency of sample [Hz]” to 20 in the popping up window.

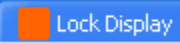



Once “Start rotation” is clicked, the “Spin ON/OFF” button on the BSMS panel will be lit flashing first and then steady.

- (**Very Important, so be patient here**) Click on **Shim**  and choose “Manual shim using “hard” shim keyboard” followed by **OK**.







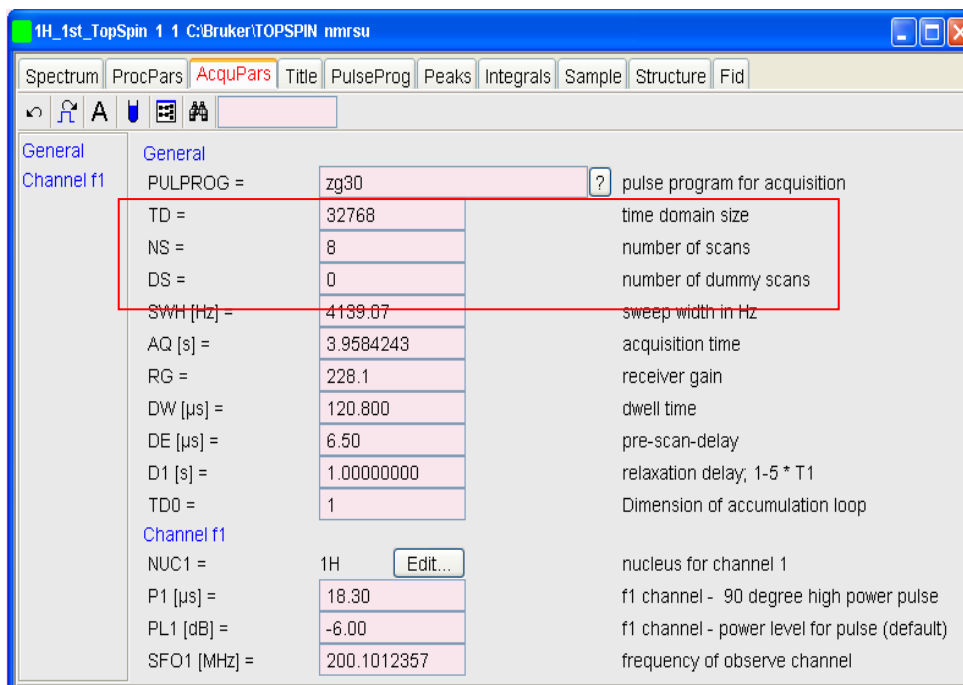
- ❖ Make sure the lock display is visible. If not, bring it up by clicking on  at the bottom of screen or on the orange square at the right hand side of the upper toolbar.
- ❖ Press “Z” button and turn the wheel on the BSMS panel to shim Z. The higher the lock signal, the better the shim. When the lock signal is at its highest, switch to Z2.
  -  If lock signal is out of window, on the panel press the “**Lock Gain**” button and reduce the value by turning the wheel to bring lock signal down to the visible region of lock display.
- ❖ Press “Z2” button and turn the wheel on the BSMS panel to shim Z2. The higher the lock signal, the better the shim. When the lock signal is at its highest, switch to Z.
- ❖ Repeat Z and Z2 shimming above for 3 to 5 rounds until the lock level is optimized.
- ❖ Press “**Standby**” button on the BSMS panel to deactivate the wheel, so as to prevent accidental change of shims.
- ❖ Click on **Close** in the Shim Info window.





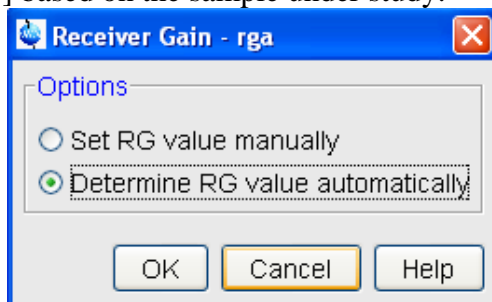
- ❖ See [Appendix C. Shimming the Magnetic Field --- VERY IMPORTANT](#) for shimming details.

6. Click on **Acquisition Pars**  [ased] for modification of parameters. Set “TD” = 32k, “NS” = 8 and “DS” = 0 for simple <sup>1</sup>H experiments for practical samples.



 Please refer to [Appendix H: Spin Dynamics and Relaxation](#) for simple spin dynamics of one-pulse FT-NMR.




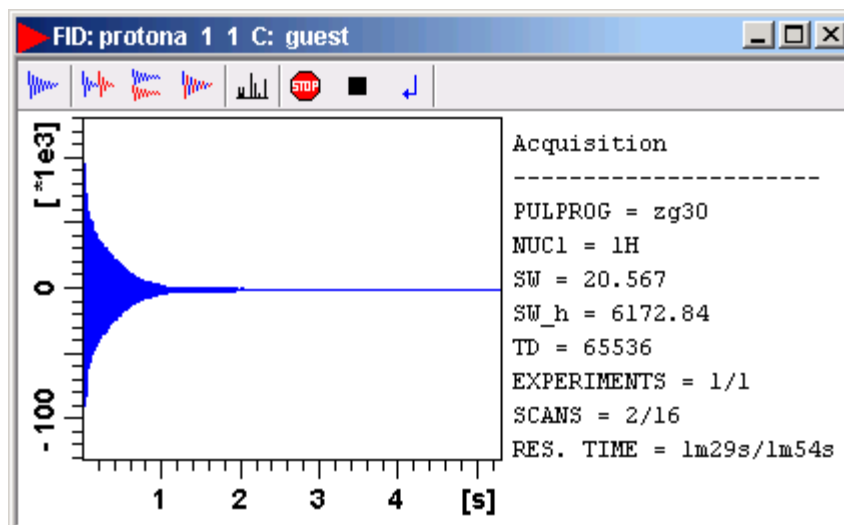
7. Click on **Prosol Pars.**  to read the prosol (instrument dependent) parameters (Power levels, e.g. “*PLI*”, and Pulse lengths, e.g. “*PI*”).
8. Click on **Receiver Gain**  and set Options to “Determine RG values automatically” [*rga*] based on the sample under study.






After **OK** is clicked, wait for message “rga finished” in the status bar of TopSpin window to occur, indicating completion of rg setting.


9. Click on **Start Acquisition**  [*zg*]. (: If a warning message occurs, make sure to start acquisition in the right dataset). The FID display window below will appear along with some status parameters.


 If parameters are not shown, right-click in the FID window, choose “Display Properties”, check “Status Parameters” and click on **OK**.




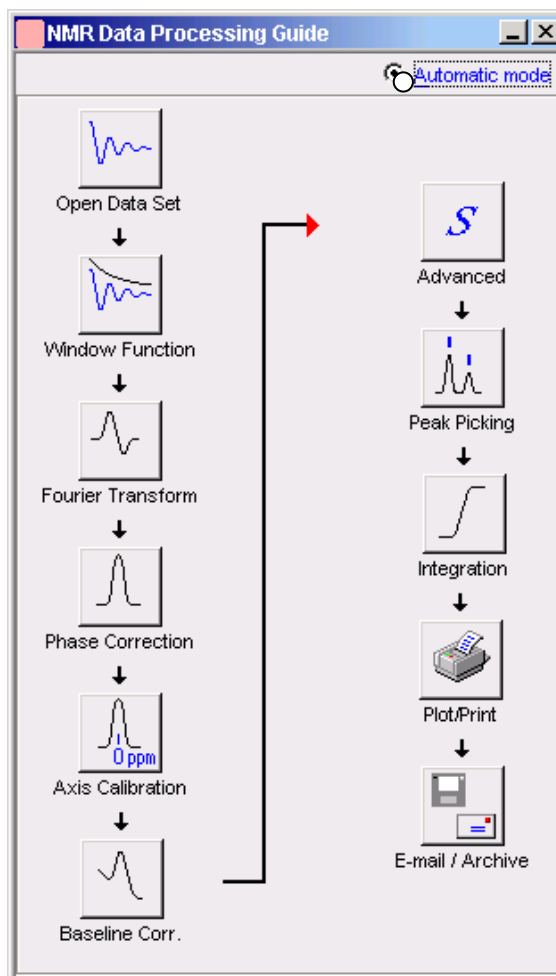
Three of the buttons near the top of the window may be used to:

-  Stop the acquisition [*stop*] with the data in buffer discarded
-  Halt the acquisition [*halt*] with the data in buffer saved to disk
-  Close the FID window

 To monitor the acquisition status, just look at the Status Parameters to the right of FID. When Res. Time = 0, acquisition is completed and it is time to process the raw data.

Click  to close the FID window once acquisition finishes.


10. Click on **To Processing**  to start the Data Processing Guide, the counterpart of the Data Acquisition Guide. Follow through the steps in the window popping up.




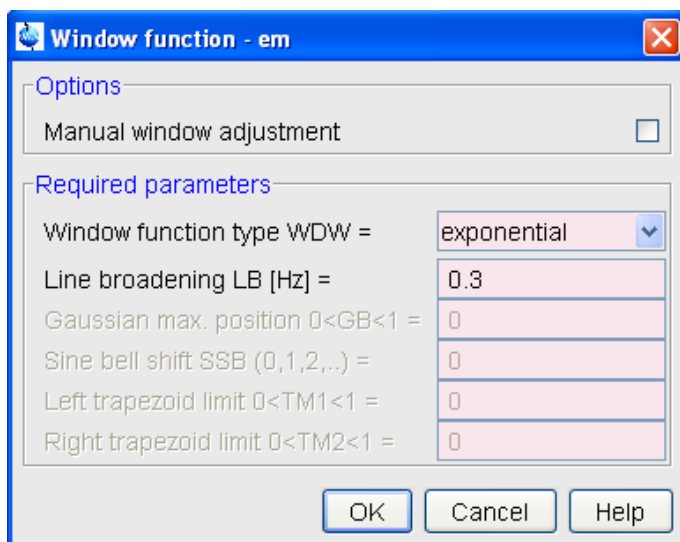
## VI. NMR Data Processing

The interactive mode, i.e. with the Automatic Mode unchecked, walks you through the data processing step by step. (See [Appendix D. 1D Data Processing Details](#)).




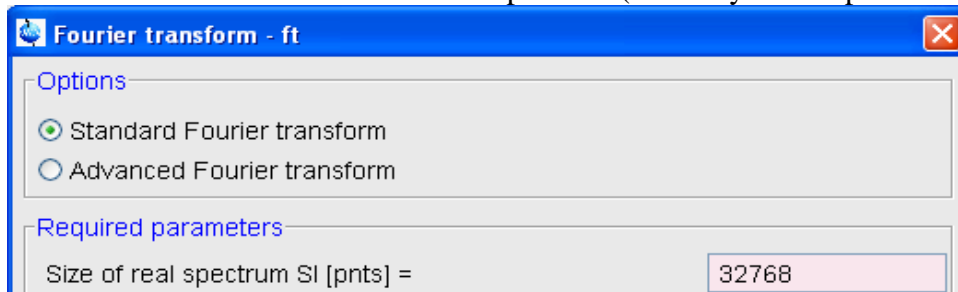
Please note that the first button **Open Data Set**  can be skipped if you process the data just acquired and shown in the active window. Otherwise, use this button to open a different dataset for processing.

- 1) Click on **Window Function**  and set “Line Broadening LB (Hz) =” 0.3 and “Window function type WDW =” exponential, followed by **OK**.

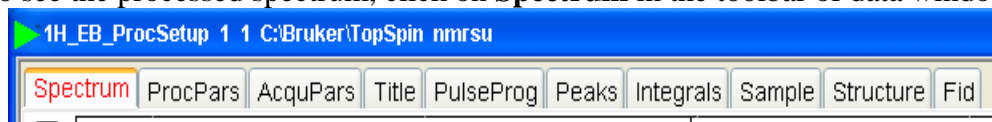



LB is a parameter to reduce noise level at the expense of resolution. The larger the LB, the better the S/N ratio but the worse the resolution. Typically “LB” = 0.3 Hz and the exponential WDW ( $\text{Exp}(-lb*t)$ ) is multiplied with the raw data FID to generate a new FID with suppressed noise. [See [Appendix E: LB and Window Function](#) for details]

- 2) Click on **Fourier Transform** , and set  
 “Standard Fourier Transform”  
 “Size of real spectrum SI = 32768”  
 and then click on **OK** to convert FID to spectrum (normally out of phase though).






To see the processed spectrum, click on **Spectrum** in the toolbar of data window.

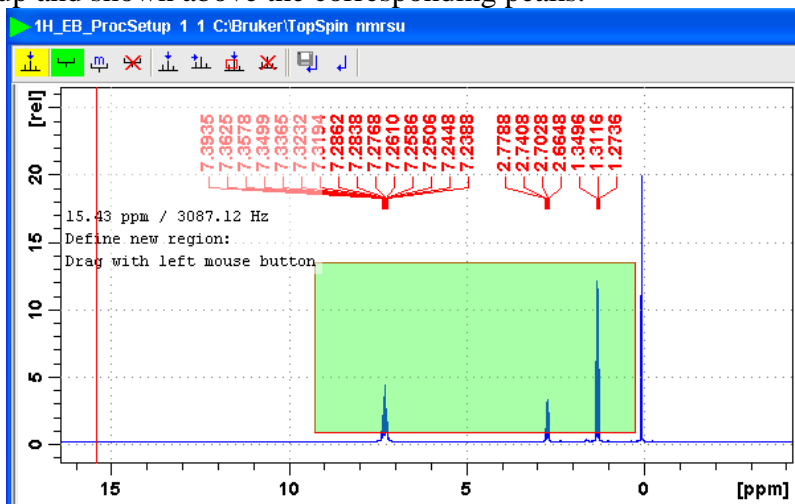



- 3) Click on **Phase Correction**  and check “Automatic Phasing” followed by **OK** [*apk*]. A phase corrected spectrum is obtained. (See [Appendix D. 1D Data Processing Details](#) for manual manipulation of phase correction)
- 4) (**Optional**) Calibration: this defines the position of 0ppm, which is the chemical shift of TMS. Since the spectrum is referenced already to the solvent, chemical

shift calibration is not necessary. However, if you do want exact calibration to


TMS, click on button **Axis Calibration**  and follow the on-screen instructions.


- 5) Click on **Baseline Corr.**  and check “Auto-correct Baseline using Polynomial” [*abs*] followed by **OK** to flatten the baseline of spectrum.
- 6) (**Optional**) **Advanced** : for multi-spectra display, add/subtract, and deconvolution (See [Appendix F: Multi-Display](#) for details).
- 7) Click on **Peak Picking** , choose “Define regions/peaks manually, adjust MI, MAXI”, and click on **OK**. In the data window, drag with the left mouse button a box (green) which defines MI (min. intensity), MAXI (max. intensity), by the bottom and top sides of the box, respectively, and chemical shift limits by the left and right sides. Peaks falling in this box both intensity- and shift-wise will be picked up and shown above the corresponding peaks.

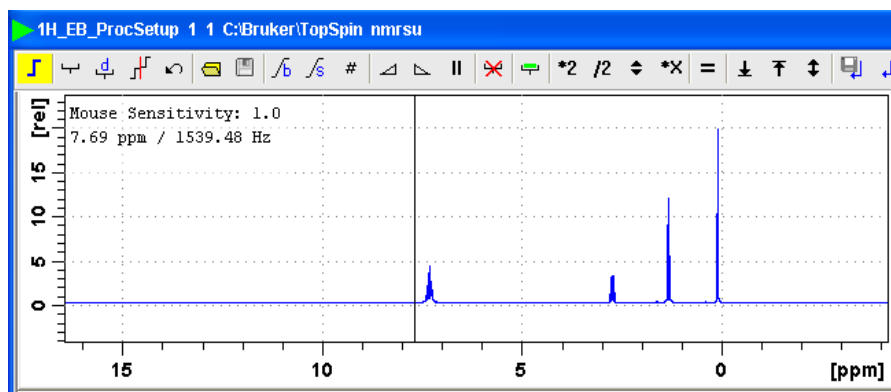



To modify the box, click on  and drag sides or corners.

To delete the box, click on .




To save the peak picking values, click on .

- 8) Click on **Integration**  and choose “Define Integral Regions Manually” followed by **OK**. An integration window appears with the corresponding toolbar displayed at the top of spectrum:







 If you see integration labels and values in the window opened up, delete them first by following the procedure “How to delete integral regions” below.


How to Define Integral Regions and do Integration interactively:


- a. Click the following button (button turns green):  
 Define integral region interactively
- b. Put the red cursor line on one side of a peak or multiplet.  For accurate results, make sure the integration starts and ends at the baseline, and D1 is long enough).
- c. Left-click-hold and drag the cursor line to the other side of the peak or multiplet.
- d. Do step 2 and 3 for all regions to be defined and integrated.
- e. Click the  button to save integration and leave the integration mode.

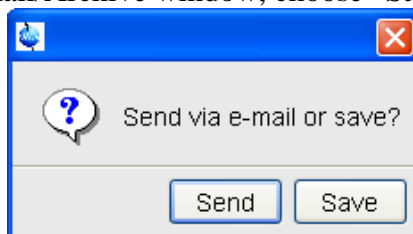
How to delete integral regions:

- To delete all integral regions, click  to Select/Deselect all integral regions, and click  to Delete selected integral regions from the display
- To delete a single region, right-click on the region to be deleted and choose “Select/Deselect” and then click on the delete button .

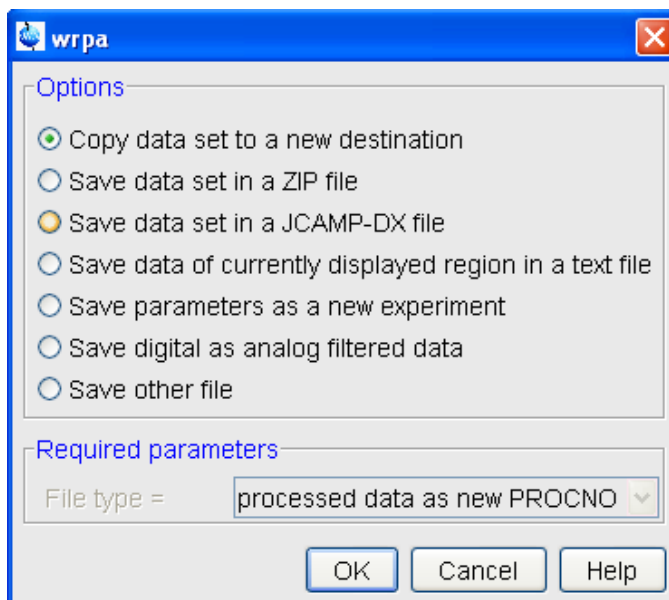
- 9) Click on **Plot/Print**  and check “Print Active Window [*prnt*]”, or check “Print with Layout – start Plot Editor [*plot*]”, followed by **OK**. The former will print what you see in the TopSpin data display window while the latter uses the more sophisticated PlotEditor program for more controlled printing. (See [Appendix G: Plot Editor](#) for details.)

 To Export Data, go to *File* → *Export ...*, specify a folder in the “Look in” box, give a filename in the “File name” box with one of the “Legal File Extensions” shown in the “Files of type” box (e.g. 1H\_5pEB.png), and click on the “Export” button.

- 10) (Optional) Click on **E-mail/Archive**  to send the NMR data by email or archive it for storage. Since emailing data is prone to problems, archiving data is a better choice. In the E-mail/Archive window, choose “Save”



Then select one of the choices in the next window (e.g. “Copy data set to a new destination” in this example or “Save data of currently displayed region in a text file”, which is a portable **ASCII** file) followed by **OK**



Change DIR to the destination of your choice (e.g. E:, a flash drive) and click **OK**.

## VII. Finishing up



**YOU ARE NOT FINISHED WITH THE SPECTROMETER UNTIL YOU DO THE FOLLOWING.**

- Make sure the D<sub>2</sub>O sample is in the first slot of sample changer.
- Press “Lift ON/OFF” button on BSMS panel to eject your sample and rotate the D<sub>2</sub>O sample to the loading position.
- Hit “Spin ON/OFF” button on BSMS panel to load the D<sub>2</sub>O sample. Sample spinning should start automatically.
- Type “lockd2o” in the command line to lock the spectrometer on D<sub>2</sub>O and reset shim.
- Quit lock window by pressing “Quit” or clicking X of the lock window.



- f. Exit TopSpin by clicking the X at the top-right corner of the software.
- g. Press “**Standby**” button on the BSMS panel if necessary.
- h. Logoff your account: click Start (bottom-left corner) and choose logoff followed by “Yes”.
- i. **Important:** On the logsheet, record your stop and duration times, and the spectrometer status. Report problems if any.

## VIII. Acquiring and Processing a 1D $^{13}\text{C}$ Spectrum ( $^1\text{H}$ Decoupled)



Normally, an 1D  $^{13}\text{C}$  experiment may take hours or days depending on concentration (Recommended concentration is at least 50 mM).

1. Go to *Spectrometer* → *Data Acquisition Guide* in the menu bar of TopSpin.
2. Click on **New Experiment** in the Guide window (see Procedure for  $^1\text{H}$  NMR).
3. In the New ... window, choose one of the standard parameter files for  $^{13}\text{C}$  experiments below:


Par file	Spectral information
C13APT	CH+CH3 Positive, C+CH2 Negative (or vice versa), Intensity not quantitative
$^{13}\text{C}$ PD	all carbons POSITIVE, for quantitative analysis
C13DEPT135	CH+CH3 Positive, CH2 Negative, C Gone, Intensity not quantitative
C13DEPT45	CH+CH2+CH3 Positive, C Gone, Intensity not quantitative
C13DEPT90	CH+CH3 Positive, C+CH2 Gone, Intensity not quantitative



The first two experiments are the most popular ones. If only the carbon types are of interest to you, use C13APT. If carbon signals are going to be quantified, choose C13CPD.

4. Click on **Lock** in the Guide if you have not done so (see Procedure for  $^1\text{H}$  NMR).
5. Click on **Sample Rotation** if spinning is not on (see Procedure for  $^1\text{H}$  NMR).
6. Click on **Shim** and perform manual shimming (see Procedure for  $^1\text{H}$  NMR).
7. Click on **Acquisition Pars** (see Procedure for  $^1\text{H}$  NMR) for modification of parameters. Add “\_wr” to the name of PULPROG, set TD = 16k, NS = 64, DS = 0, and L31 = 2000 for  $^{13}\text{C}$  experiments.
8. Click on **Prosol Pars** (see Procedure for  $^1\text{H}$  NMR).
9. Click on **Receiver Gain** (see Procedure for  $^1\text{H}$  NMR) and set Options to “Set RG values manually”. Input “16k” followed by return.



To know how long the experiment lasts beforehand, click on  in the upper toolbar.

10. Click on **Start Acquisition** (see Procedure for  $^1\text{H}$  NMR).
11. Process data (see Procedure for  $^1\text{H}$  NMR).



**A few things to note:**

- You don't have to wait until the experiment is done to process data. You can do processing as soon as the first NS scans are finished and saved to disk.

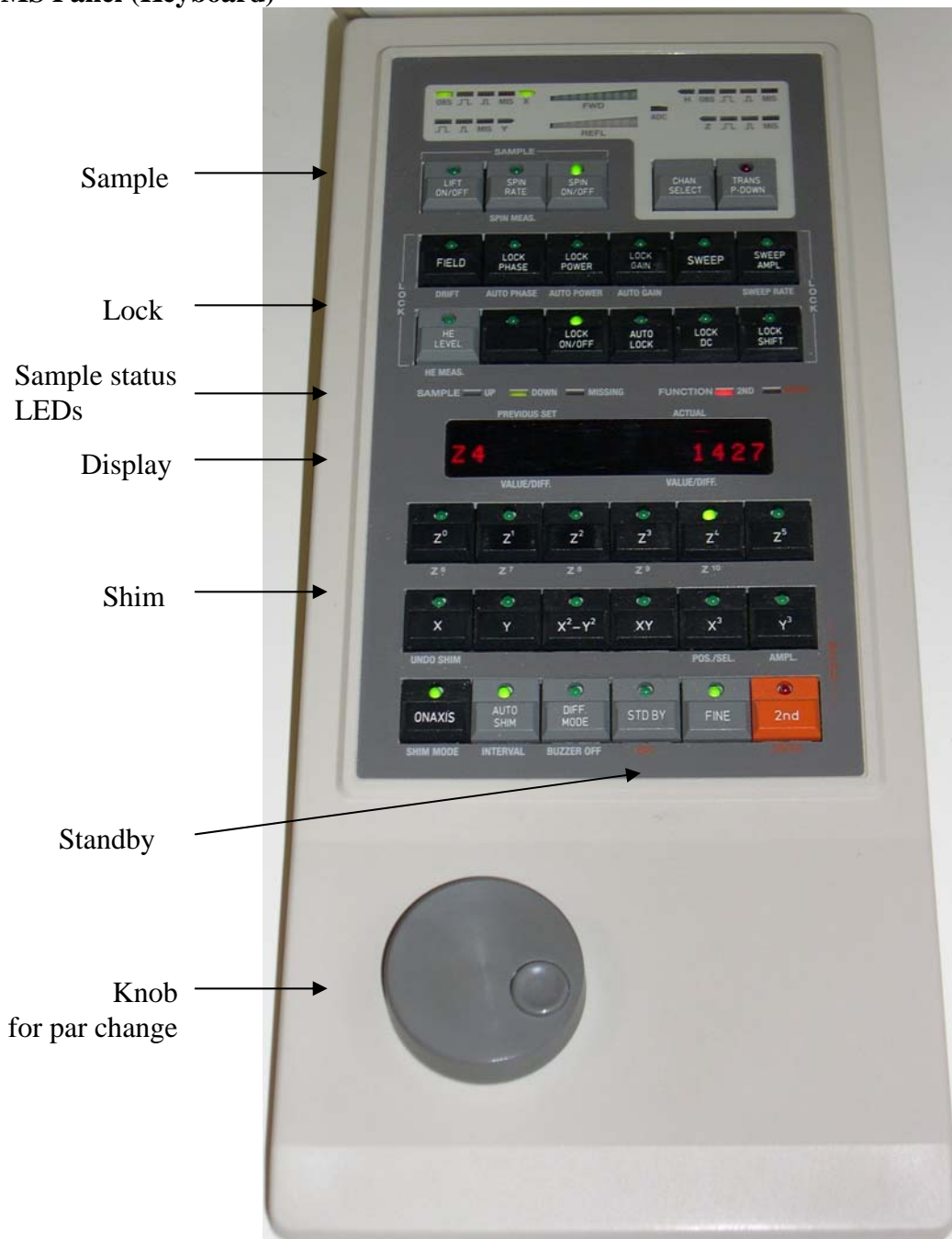
- A larger line broadening value is used for  $^{13}\text{C}$  than for  $^1\text{H}$ . Typically  $lb = 1 \text{ Hz}$ .
12. Type "**halt**" if spectrum is satisfactory or let it continue if not. The acquisition will stop by itself after  $NS*L31$  (maybe  $L30$  or  $L16$ ) number of scans are completed.
  13. For Calibration, Integration, Plotting, etc of a  $^{13}\text{C}$  spectrum, please refer to the procedure for  $^1\text{H}$  NMR.

## IX. Finishing up

(YOU ARE NOT FINISHED WITH THE SPECTROMETER UNTIL YOU DO THE FOLLOWING.)

- a. Make sure the D2O sample is in the first slot of sample changer.
- b. Press "**Lift ON/OFF**" button on BSMS panel to eject your sample and put the D2O sample at the loading position.
- c. Hit "**Spin ON/OFF**" button on BSMS panel to load the D2O sample. Sample spinning should start automatically.
- d. Type "lockd2o" in the command line to lock the spectrometer on D2O and reset shim.
- e. Quit lock window by pressing "*quit*" or clicking X of the lock window.
- f. Exit TopSpin by clicking the X at the top-right corner of the software.
- g. Press "**Standby**" button on the BSMS panel if necessary.
- h. Logoff your account: click Start (bottom-left corner) and choose logoff, followed by "Yes".
- i. **Important:** On the logsheet, record your stop and duration times, and the spectrometer status. Report problems if any.

**X. Appendices**  
**A. BSMS Panel (Keyboard)**



**B. Lock details and troubleshooting**






In the lock display window, you can view the lock signal after lock-in has been successful. At the top of the lock window the following buttons are available:



Open the user preferences window [*set*]



Toggle lock monitor mode

	Toggle lock display mode between <i>single</i> and <i>dual</i> color
	Switch grid mode between <i>both</i> , <i>vertical</i> , <i>horizontal</i> and <i>off</i>
	Make the lock display external
	Put focus into TOPSPIN window
	Close the lock display window

#### Troubleshooting:

- iv. Problem: “**Lock ON/OFF**” won’t stop flashing after [*lock*]
- v. Possible reasons:
  1. You started with a bad shimming file
  2. Your sample is too concentrate
  3. Too little deuterons in your solvent
- vi. Solutions:
  - for 1. Type [*rsh*] in the command line to read in a good shimming file and lock again
  - for 2 & 3. Do manual locking as below:
    - Press “**Lock ON/OFF**” to turn off lock (LED off). At this point, you may see a noisy and weak oscillatory lock signal.
    - Hit “**Lock Gain**” and rotate the wheel on BSMS panel to increase lock signal amplitude until the signal fits the whole lock window.
    - Then press “**Lock ON/OFF**” to try to lock spectrometer. The LED should stay solid after brief flashing if spectrometer locks (the oscillatory signal becomes a flat sweeping line). If not,
    - Hit “**Lock Power**” and rotate the BSMS wheel to increase lock power. The lock signal will increase accordingly and may be saturated, i.e. the signal will not increase anymore with power. Remember: if the saturation happens, bring back the power by 2~3 units in reading.
    - Then press “**Lock ON/OFF**” and immediately increase “**Lock Power**” until the spectrometer is locked, where the “**Lock ON/OFF**” LED should stay solid and the oscillatory signal becomes a flat sweeping line. Avoid saturation stated above.
    - If you still have trouble, please ask Jerry for help/debugging.

### C. Shimming Magnetic Field --- **VERY IMPORTANT** (!!! be patient and take your time here !!!)



No keys on BSMS panel will response if “lock finished” is not seen in the status bar at the bottom of the TopSpin window.

Normally you only need to work on Z & Z2 for practical samples. There are two ways for shimming:

1. Manual shimming:
  - a. Press “Z” button, turn the BSMS wheel to optimize lock signal (i.e. lock signal won’t go higher), the higher the better.
  - b. Do the same thing for “Z2”.
  - c. Repeat steps a & b above alternatively for a few times until the lock signal doesn’t go higher any further.
  - d. Acquire a <sup>1</sup>H spectrum to check lineshape of signals. If symmetric without any foot or shoulder, you are done with shimming. If not, do steps e-f-g below.
  - e. Change “Z3” in either direction and repeat steps a-b-c above. If the lock signal is higher than that prior to Z3 adjustment, keep changing “Z3” in the same direction and repeat a-b-c until lock signal is optimized. Otherwise, reverse the direction of “Z3”.
  - f. As for “Z3”, change “Z4” and repeat steps a-b-c-e above.
  - g. If necessary, repeat steps a-b-c-e-f several times until lock level doesn’t go any higher.
2. Auto shimming:



It is good only for keeping and gradually improving shimming over a long period of time. Since the process is really slow, it doesn’t worth of doing for just <sup>1</sup>H experiments. It is useful though when both <sup>1</sup>H and <sup>13</sup>C NMRs are required. Do <sup>13</sup>C first with Auto Shimming turned on at the same time and then do <sup>1</sup>H once <sup>13</sup>C finishes. By the time <sup>1</sup>H starts, shimming may have be improved quite a bit.

- Do rough manual shimming first.



If no prior manual shimming, it will take at least a couple of hours to reach a reasonable shimming level. Be patient and take your time on manual shimming before proceeding.

- Press “AUTO SHIM” button or type “tune z1234” in the command line if the button does not work.


## D. 1D Data Processing Details

- a. Process data
  - i. Set line broadening  
type “*lb*”, then enter a number  
typically 0.3 Hz for <sup>1</sup>H
  - ii. Type “*ef*” (ef = em + ft)  
This command baseline corrects, exponentially multiplies, and Fourier Transforms the data. You will now be switched over to the spectrum window (frequency domain) and see an unphased NMR spectrum.




[Appendix E: LB and Window Function](#)


b. Phase the spectrum (make all peaks absorptive)

- i. Click on  button (in the upper toolbar) and the toolbar above your spectrum should look like this:




- ii. Move the red cursor line to the tallest peak, right-click there, and select Set Pivot Point to mark the peak with a vertical red line, indicating a pivot point to be used by Ph1. Right-click again and choose "Calculate Ph0". The tallest peak will now be phased roughly right.
- iii. Click and hold on **0** (zero order phase, used when the phase of peaks is frequency-independent). Dragging the mouse will change the zero order phase. Make the tallest peak exactly absorptive.
- iv. If other peaks are still not leveled on both sides, Click and hold on **1** (first order phase, the phase of peaks is directly proportional to their frequency positions relative to the pivot point set above). Dragging the mouse to fix their phase. Many times this will also straighten out the baseline.
- v. Click on  to save the phase values and leave the phase mode.
- vi. If you want to process the same data again after you have done phasing, use "*efp*" (efp = em + ft + phase) instead of "*ef*", so you don't have to do "*phase*" again.


c. Calibrate the spectrum (if necessary)

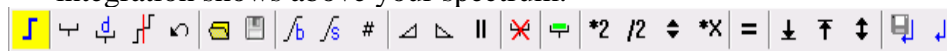
- 1) Locate the solvent or TMS peak of known chemical shift, and expand its nearby region by click - hold - dragging across the peak.
- 2) Click on  button.
- 3) Go to the top of the reference peak chosen in 1) and enter the chemical shift value of that peak (the solvent table on desk may help).




d. Integrate peaks










It might be easier to zoom in on a small region before integrating and use the  buttons to move around during integration for better differentiation of peaks.

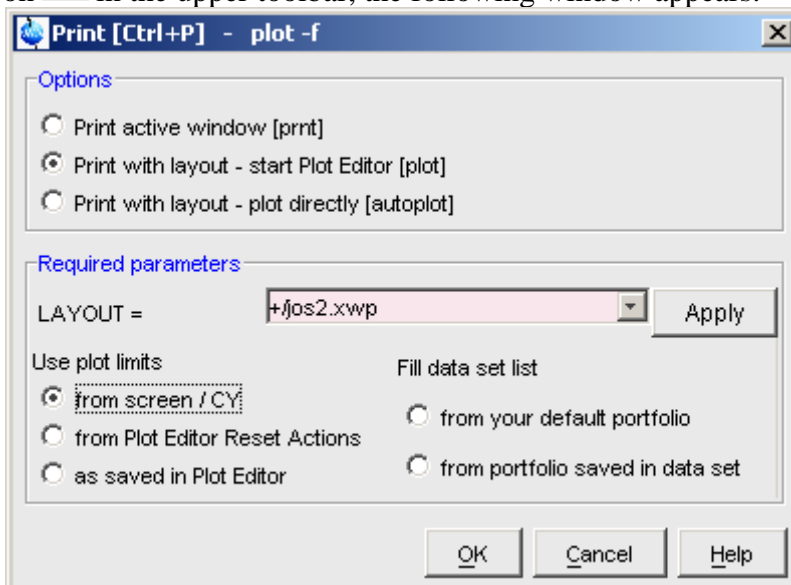
- 1) Type "*abs*" to flat the baseline.
- 2) Click on  to enter the integration mode. The toolbar below for integration shows above your spectrum:



- 3) Click on  and then on  to delete all the integrals originated from step 1).
- 4) Click on  for manual integration.
- 5) Drag from one side of a peak to the other to integrate this peak.
- 6) Repeat step 5) for each peak or group of peaks of interest.

- 7) For more accurate integration, click – hold on  and move the mouse to flat the left tail of an integral and use  for the right tail.
- 8) Once done with all peaks of interest, click on  to save the integration values and leave the integration mode.
- e. Peak-picking
- Click on  to start peak picking and show the corresponding toolbar:
- 
- Draw a box to define the peak picking range (vertical sides of the box) and intensity limits (horizontal sides of the box) by placing the cursor at the click – holding the LMB and dragging from the top left corner of the box towards the bottom right corner.
  - Peaks falling in that box are automatically picked up and labeled.
  - Click on  to save the chemical shift values and leave the peak picking mode.
  - If necessary, click  to delete a peak picking region
- f. Plot Spectrum

Click on  in the upper toolbar, the following window appears:

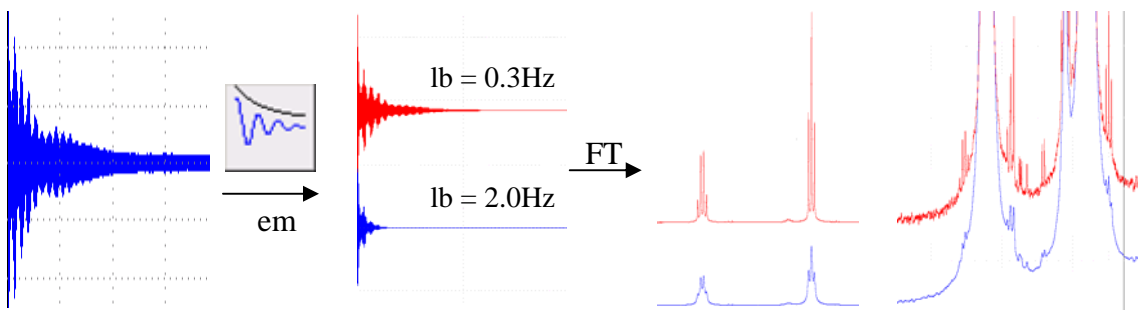


Turn on the first radio button to print the active window or the 2<sup>nd</sup> button for the more powerful PlotEditor. (See [Appendix G: Plot Editor](#) for details.)

- g. To save spectrum as an electronic file:
- 1) Insert a flash drive or a ZIP disk into computer.
  - 2) In TopSpin or Plot-Editor, go to “File” → “Export” →
  - 3) Specify the destination folder (e.g. where the flash drive or Zip is), filename and file format (e.g. <sup>1</sup>H\_NMR.png, .jpg, .tif, ...) → **Export**.
  - 4) Unmount flash drive or ZIP.

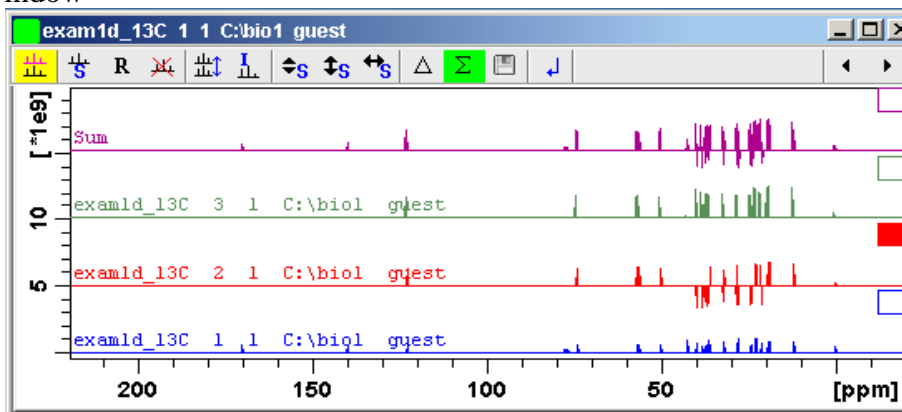


## E. LB and Window Function



## F. Multi-Display

1. Read a 1D dataset by right-clicking on it and choosing “Display” in the browser/portfolio.
2. Click the button in the upper toolbar. The data window will switch to multiple display mode.
3. Add a dataset as follows:  
Left-click-hold the dataset in the browser/portfolio and drag it into the data window

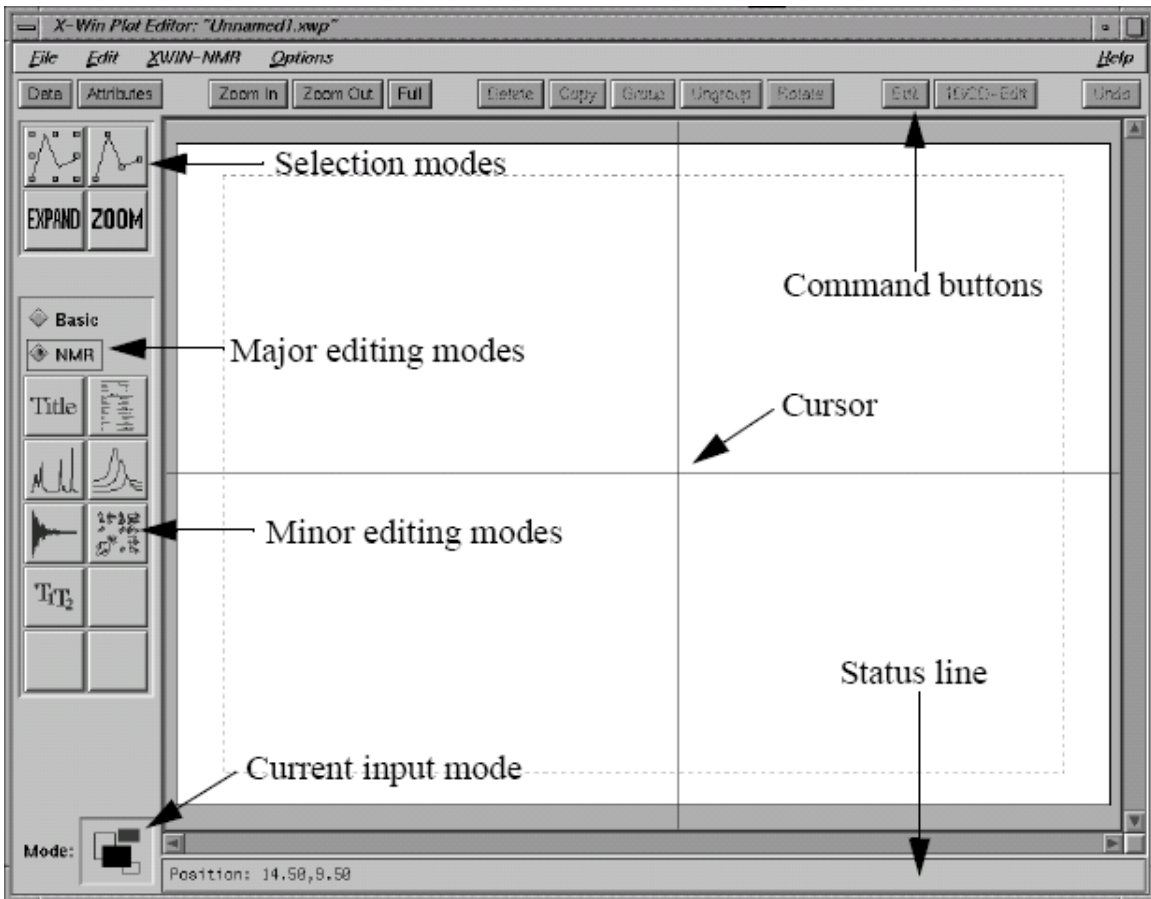


Use the toolbar of the multi-display window for further manipulation and move the mouse cursor over each button for help or refer to the TopSpin Users’ Guide for more details.

## G. Plot Editor

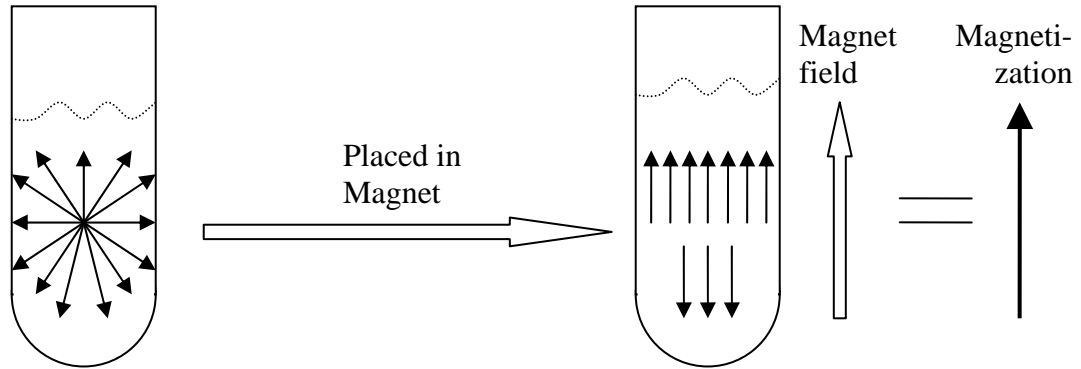
In the *File* → *Print* window, if you select “Print with layout - start Plot Editor [*plot*]” option and click **OK**, the Plot Editor will be started. This option is equivalent to entering *plot* on the TOPSPIN command line.

This is a very nice and user-friendly program. You are referred to the Plot Editor manual of TopSpin for more details and encouraged to use it often for more controlled printouts.

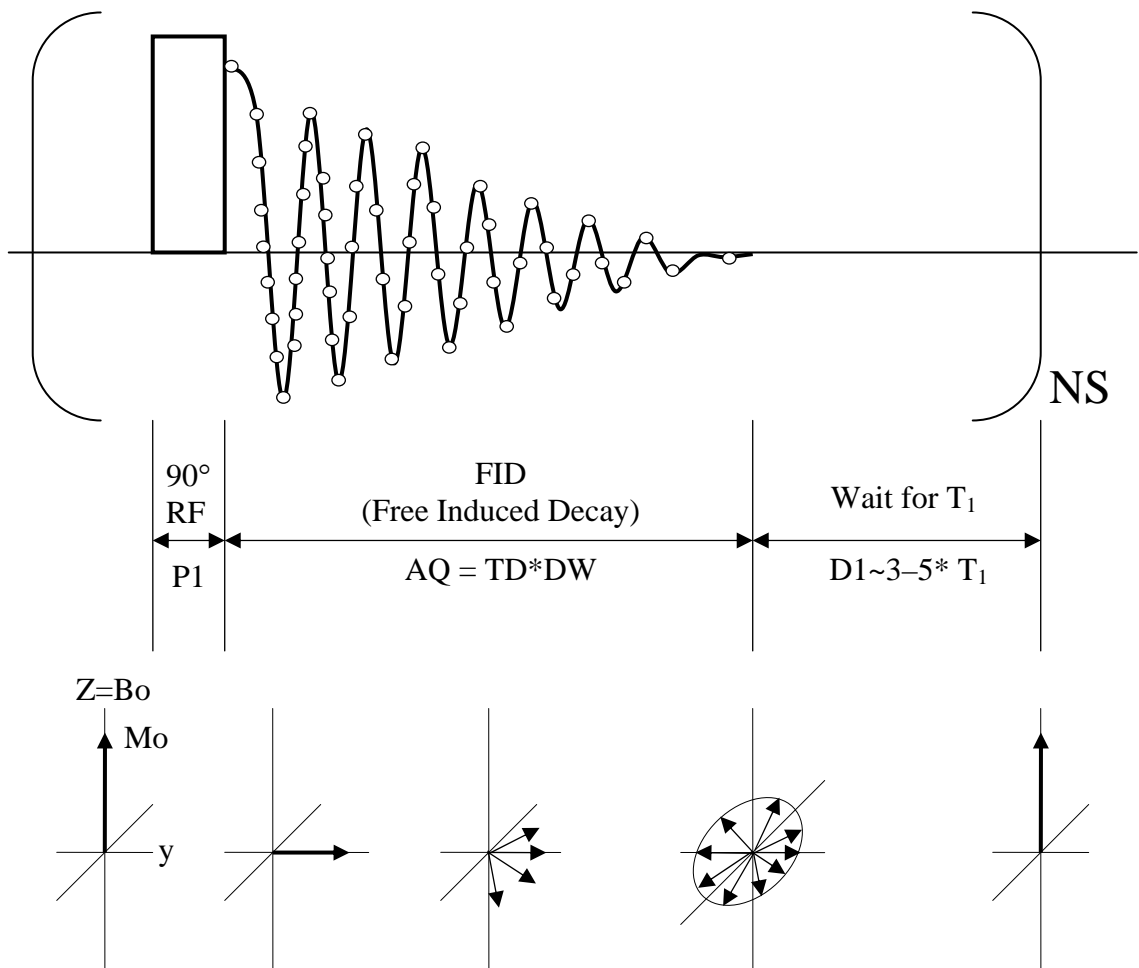


## H. Spin Dynamics and Relaxation


### Alignment of nuclei in magnetic field



### Excitation, Acquisition and Relaxation

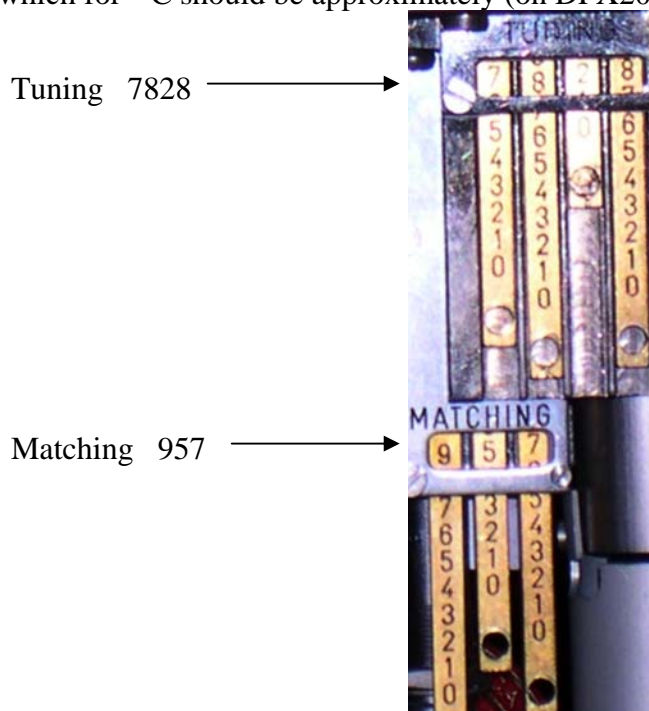



## I. Probe Tuning and Matching

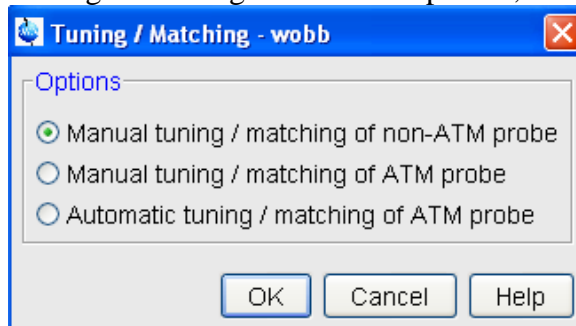
 When you do NMR on a nucleus X other than  $^1\text{H}$  and  $^{13}\text{C}$  (e.g. X =  $^2\text{H}$ ,  $^{19}\text{F}$ ,  $^{29}\text{Si}$ ,  $^{31}\text{P}$ , or ...), you will need to tune the probe. You do not normally need to do this when performing  $^1\text{H}$  or  $^{13}\text{C}$  NMR.

### Steps

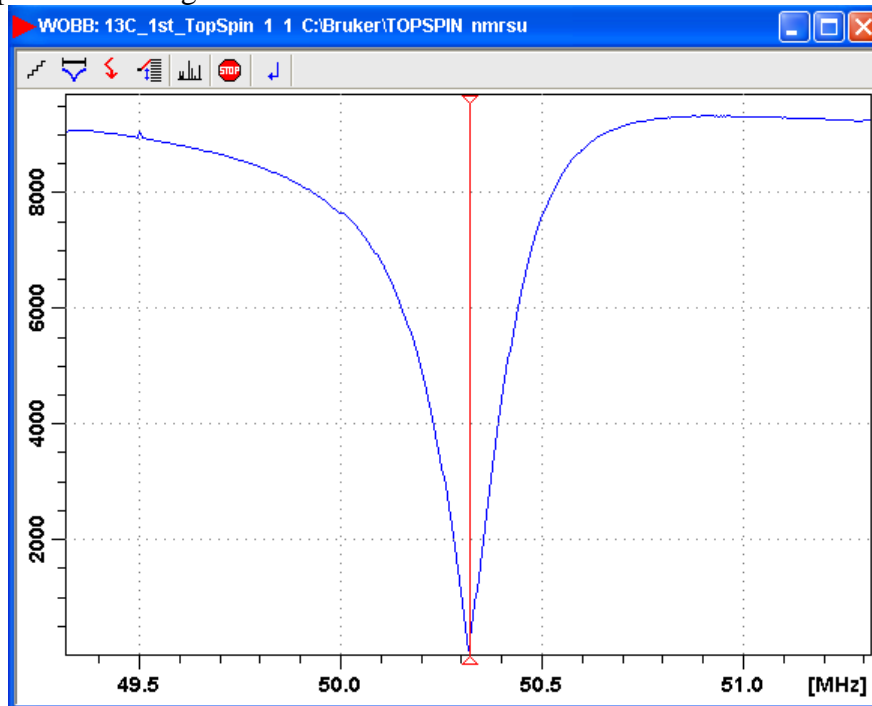
- Check the numbers in the windows of tuning and matching sticks of the probe, which for  $^{13}\text{C}$  should be approximately (on DPX200 MHz)



- If the values currently on the tuning sticks are within the range given in step a, the probe is tuned to  $^{13}\text{C}$ . But it is always a good idea to check if the tuning is exact.
- Click on **Probe Tune/Match**  in the **Data Acquisition Guide** and select “Manual tuning / matching of non-ATM probe”, followed by **OK**.



After ~20s, you will see a WOBB window showing the tuning/matching curve, the horizontal position of which corresponds to tuning and the depth to matching.



The tuning curve should be aligned on the screen with the central redline and should reach all the way to the zero line of Y axis. If it doesn't look this way, then the probe isn't tuned.

d. Go to the probe.



This is a piece of equipment which goes into the magnet from the bottom. It has cables and hoses attached.


e. With the supplied tool, pull/push with fine control the tuning sticks so that the curve aligns with the vertical red line and the matching sticks to make the curve reach all the way to the zero line of Y axis. Go back and forth between tuning and matching for optimization.




You can also look at the preamplifier box called HPPR (a small box with cables next to the magnet) and minimize the number of LEDs lit on the horizontal (tune) and the vertical (match) LED arrays, normally 3 green LEDs lit for match and 1 green LED (and maybe a yellow one) lit for tune.



f. Tune and Match  $^1\text{H}$  channel:

After the  $^{13}\text{C}$  channel is optimized, click on  to switch to  $^1\text{H}$  and wait until the  $^1\text{H}$  curve occurs (takes ~20s). Adjust the Tuning and Matching Rods for  $^1\text{H}$  (located on the opposite side to the  $^{13}\text{C}$  tuning/matching sticks. The rod labeled with T is for tuning and M for matching) to optimize  $^1\text{H}$ .

g. Then Click on  to quit the probe tune/match process.

## J. Online NMR Book and Bruker NMR Encyclopedia

- 1) NMR Book: <http://www.cis.rit.edu/htbooks/nmr/>  
Introduction to NMR concepts and practical issues.
- 2) NMR Guide & Encyclopedia: <http://www.bruker.de/guide/>  
All you want to know about NMR.

## K. Requirements for Access to the MRL NMR at CNSI

You have to pass the mini quiz within one month after training in order to be qualified for access to the NMR facility of MRL, which includes:

- Key Card for Lab & Building (see Sylvia in Rm. 2066G for Authorization Form)
- Web Scheduling Account (email Jerry for a setup appointment)

- NMR Account (email Jerry for a setup appointment)

These requirements apply to both on- and off-campus users.