Seton Hall University

200 MHz NMR

SOP manual

NMR Information:

Varian Gemini 2000 NMR
^{1}H Frequency = 200 MHz
Original purchase date: 1989
Donated from Merck & Co., Inc., 1998
Software upgrade: 2008

To schedule time or check availability on the 200 use:
http://academic.shu.edu/chemistry/calendars/NMR-200/

Version: 14 January 2012

<u>NMR Cooperative Maintenance and Use Program:</u> ***anyone who wishes to use the either NMR must participate in a training program, contribute to the maintenance of the instruments/facility and contribute to the advancement of the instrument.***

Each Time:

1. **Check when the NMR was last filled.**
2. **Measure N_2 and He boil off and fill levels and record in white binder.**
3. **Record your experiment in the hardbound notebook**
4. **Run experiment!**
5. **Return Dummy sample, lock, shim, take ^1H and make sure NMR is left in good working condition.**

Routine problems: consult your faculty advisor or an expert user. If problem persists, contact Prof. Sowa (200) or Prof. Murphy (500).

Emergency problems contact: Prof. Sowa (973)738-2886, and/or, Prof. Murphy (973)714-1715.

Table of Contents:

Safety Precautions

Hazards associated with maintenance and use of an NMR instrument are described in the University's Chemical Hygiene Plan. In summary, there are three main hazards to NMR experimentation:

a) exposure to strong magnetic fields

The NMR is a strong, superconducting magnet. If you have a pacemaker, it is suggested that you stay beyond the 10 gauss line which is at least 10 feet away from the magnet. Generally all objects that are easily attracted to a magnet such as metal tools, keys, chairs should be kept beyond the 5 gauss line which is at least 5 feet away from the magnet. In addition, credit cards with a magnetic strip, computer drives, computer disks and flash drives should be kept at least 5 feet away.

b) exposure to cryogenic inert gasses of helium and nitrogen

Cryogenic gasses are hazardous for two reasons. First, the extreme cold is enough to immediately freeze skin tissue and can have the effect of a severe burn. People who routinely fill the NMR with liquid nitrogen should be aware of this hazard and wear safety glasses, gloves and protective clothing. Second, these gasses displace air. In the case of a sudden release and accumulation of these gasses, suffocation can occur. The room is equipped with an oxygen sensor which monitors oxygen levels in the room and beeps if the levels are unsafe. All users should be aware of the potential of a magnet quench. This is a catastrophic event that causes the helium and nitrogen gas to suddenly release from the magnet. It may preceded by a loud bang. If this event occurs, immediately evacuate the room and notify the Lab Safety Officer, David Edwards and any of the faculty in charge of the NMR.

c) NMR tubes

NMR tubes can easily break in one's hands when they are loaded into the spinner. Use proper caution when you place the tube into the spinner. Tubes that are cracked at the rim are especially susceptible to breakage and it is recommended that these be discarded. Tubes may also break due to chemical events inside the tube such as pressure build-up due to gas release or a reaction exotherm. Thus, be aware of the chemical hazards associated with your experiment.

Style and Contribution Comments

This manual is intended to assist all users of the 200 MHz NMR with helpful procedures or routine and advanced experiments. Contributions and updates are welcome!

Style comments:

Acquire underline means that a button needs to be selected in either the upper or lower menu bar.

nt=4 italics means that a command needs to be entered in the command line.

Contributions:

Please submit your typed contributions to John Sowa so that they may be entered into the master manual.

You may also directly write hand-written contributions into the manual and these will be periodically checked and updated.

Sample Preparation

A) NMR tubes:
a) *Caution! Do not use tubes that are cracked or chipped, especially at the top. These have a tendency to break while inserting or ejecting into the NMR probe resulting in contamination of the probe.*
b) Approved NMR tubes are carried by the SHU stockroom. Use a tube that is rated for the spectrometer frequency as follows (http://www.sigmaaldrich.com/analytical-chromatography/spectroscopy/learning-center/nmr-spectroscopy/nmr-tubes.html):

200 MHz: Wilmad (506-PP, 507-PP, 528-PP)
500 MHz: Wilmad (528-PP, 535-PP, 545-PP)

For New ERA NMR Tubes (http://newera-spectro.com/NewEraNMRcatalog.pdf):

MHz	General Application	Sample Tubes
100 - 300	Routine organic chemistry Educational applications	NE-L5-, NE-L7S-
300 - 400	Routine organic chemistry Educational applications Routine synthetic chemistry research High throughput	NL-ML5-, NL-MP5-
400 - 500	Routine synthetic chemistry research High throughput	NE-HL5-, NE-HP5-
500 - 700	Organic chemistry research Metabolic mixture analysis (biofluids) High throughput	NE-UL5-, NE-UP5-
700 - 900+	Structural biology, Metabolic analysis Multi Purpose research	NL-SL5-, NL-SP5-

c) A dry and proton free NMR tube is helpful for ^{1}H NMR analysis. Rinse a clean NMR tube with a few drops of D_2O. This will exchange any surface –OH groups with deuterium. Place tube in an oven set at 120 – 130 °C for at least 1 h. Remove the tube from the oven and immediately cap with a rubber septum or a polyethylene cap until the tube has cooled to room temperature.

B) Sample amounts:
1) You only need 0.75 mL of a suitable deuterated NMR solvent. Don't waste expensive solvent!
2) ~ 5 mg is needed for a routine ^{1}H NMR spectrum.
3) ~ 30 mg is needed to a routine ^{13}C NMR spectrum.

C) Deuterated solvents:
The SHU stockroom stocks most of the common NMR solvents. For organic compounds, $CDCl_3$ and acetone-d_6 are the least expensive solvents. Sometimes, $CDCl_3$ can be difficult to lock. Solvents stored on 0.75 to 1.0 mL ampoules are the most are quite convenient to use as there purity and dryness is very reliable. Solvents stored in larger, screw capped containers should be stored over molecular sieves and housed in a desiccators to maintain dryness.

D) Filtering the deuterated solvent or NMR solution through an HPLC filter may also result in better resolved spectra.

To schedule NMR time, use our internet-based NMR scheduler at the following URL:

http://academic.shu.edu/chemistry/calendars/NMR-200/

Logon/logoff

To logon:

1) Enter your name, date and sample information into the 200 NMR notebook.
2) Check the nitrogen and helium boil off levels. Once a week, check the nitrogen fill level with the wooden dip-stick.
3) Logon: enter the username: (to be provided)
4) Password: enter the password: (to be provided)
5) In menu bar, click on icon that shows graphic of an NMR spectrum. You are now logged-on!

To logoff:

1) Write any comments/observations about performance of instrument in notebook.
2) In command line type: *exit*
3) In menu bar, click on EXIT. (Note: if the menu bare does not show, please right click on the screen background and select EXIT.)
4) You will be asked a question to save changes and complete exit routine. Click on Yes. You are now logged off.

Screen security lock:

During long-term acquisitions, the computer will go into security lock. To use the software, reenter the password.

Ejecting and Inserting NMR samples:

1) *Caring for the spinner:* A clean and well preserved spinner will ensure that all NMR tubes spin properly. Make sure your hands are clean and free of dirt and grease. Handle spinner only on the upper black band. After loading a new sample, wipe spinner clean with lint-free paper towel.
2) Press and hold EJECT button located on left leg of the magnet. Remove Dummy sample/spinner.
3) Carefully remove the Dummy sample from the spinner. Do not break the Dummy sample!
4) Carefully insert your sample tube into the bore of the spinner. When you do this, do not hold your NMR tube on the upper end as this will create too much leverage and may cause the tube to break. Hold your tube in the middle while carefully inserting it into the bore of the spinner. Wipe spinner and tube with a lint-free paper towel.
5) **Adjusting the height of the sample: You may use the gold sample adjuster (kept by the 500 NMR), use the markings on the right leg of the 200 NMR magnet (use the thicker marking on the left side because it matches the spinner the best). Note that the sample depth must not be below the indicated level. Also, the entire volume of the sample must be within the shaded region.**
6) Press and hold EJECT. While holding the EJECT button, carefully place the loaded spinner into the bore of the magnet. Slowly release the EJECT button. You will hear a healthy "kerplunck" indicating sample has been properly inserted.
7) Check spin rate. The green spin indicator light should be on and spin rate should be approximately 20 rps. If the sample does not spin, type *spin='y'* a few times until you hear the solenoid engage.
8) When your experiment is complete, return the Dummy sample to the probe. Lock, shim and take a 1H spectrum. Inspect the spectrum which is a singlet of residual H_2O. Make sure that you are leave the spectrometer in good shape for the next person!

Stuck spinner:

If the spinner has been inserted into the magnet without a sample, it will not eject. You will need to increase pressure at the main line to 50 psi and then eject. Please return the pressure to 40 psi.

Acquiring a ^{1}H NMR Spectrum

1) Enter name, date, sample information into the hardbound notebook.
2) If you are the first user of the day, you must measure the N_2 and He boil-off rate and record these in the notebook.
3) Logon and insert sample into the magnet.
4) Click on Main Menu on upper menu bar.
5) Click on Setup on lower menu bar. This will bring you to a screen where you select the nucleus and the solvent.
6) Select nucleus (1H) and solvent. Click on appropriate icon.
7) Load the latest shims. Type *rts* and enter return. Type in the most recent shim file. Currently, this file is: *bestshims*. Then type *su*. (Note: shim files may be created for other solvents: *bestshims-cdcl3*.)
8) Lock the sample. In this procedure you will adjust parameters in the instrument that will enable you to receive an NMR signal. The procedure is analogous to tuning a radio with a dial to locate your favorite radio station.
    a) Click on Acqi in upper menu bar. A purple bordered screen will appear. (Note: if the Acqi button is not present or does not respond, this is an indication that the computer and NMR console are not communicating.)
    b) Click on the LOCK and off buttons.
    c) This will bring you to a screen displaying Z0, lockpower, lockgain and lockphase.
    d) Note the initial Z0 value. The goal is to Adjust Z0 so that it forms a plateau (⎍) and the signal is steady and not noisy.
    e) While adjusting Z0, you will need to adjust the signal strength. Do this by increasing the transmitter power (lockpower) and the receiver gain (lockgain). When the lockpower signal is too high, the lock signal pulses. When this happens, lower the lockpower. If the signal is very noisy, lower the lockgain. If the signal reads > 99 %, lower the lockpower and lockgain to ~50 – 70 %.
    f) Adjust lockphase so that the horizontal part of the signal is level and the height of the signal is maximized.
    g) It is a good idea to note the Z0, lockpower, lockgain and lockphase values for each solvent.

solvent	Z0	lockpower	lockgain	lockphase
D_2O	-505			
acetone-d_6				
$CDCl_3$				
C_6D_6	-619			
dmso-d_6				

    h) Click lock on.
9) Shim the sample: In this procedure, you will adjust the homogeneity of the magnetic field. This should lead to sharp, distinct NMR signals. This technique

take considerable skill and patience; however, once you master it, you will never forgot how to do this well!

a) Click on SHIM. This will bring you to the axial shim screen.
b) Note the Z1C, Z2C, Z1 and Z2 numbers. Maximize Z1C by clicking on the +/-1 button. Maximize Z2C by clicking +/-1 button for Z2C. Go back and maximize Z1C. Next, maximize Z1 by adjusting the +/-1 or +/-4 buttons. Then maximize, Z2 in the same way. Finally re-adjust Z1 so it is maximized.
c) You may adjust Z3, Z4 as above.
d) It is occasionally necessary to adjust the non-spinning shims (X, Y, XY, etc.). However, if this must be done, turn the spinner off at the console and make appropriate adjustments.
e) For more help with shimming a good reference is G. A. Pearson on How to Shim an NMR Magnet (http://nmr.chem.uiowa.edu/manuals/Shimming-GAP-NMR-magnet.pdf, accessed 9/30/2011) is posted on the wall behind the 200.

10) You are ready to measure your sample!
11) Close the Lock and Shim screen. Click on Main Menu. Click on Acquire on the lower menu bar. Alternatively, just type *ga* and the instrument will acquire and transform your spectrum.
12) Select the Go, Wft button. The status screen will appear and at the completion of the experiment, the NMR spectrum will appear. Processing will be described in the next section.
13) Process, save (if necessary) and print your spectrum (see next section).
14) Important! Before you leave, please eject your sample, return the Dummy sample (D_2O) and lock, shim and take a spectrum to make sure the NMR is left in excellent condition for the next user.

You have now taken a ^1H NMR spectrum!

Helpful Hints for ^{1}H NMR Spectra:

#	Hint	Initial/date
1	The default value for the number of transients collected is 16. To change this value, type in the command line: *nt=x* (where x is the new value). For infinite transients, type *nt=-1*.	JRS, 7/08
2	If the peak shapes do not look good, go back and redo the shimming routine.	JRS, 7/08
3	Sweep width (*sw*) and *tof* (transmitter offset) *sw=3000* scans from -2 to +12 ppm *sw=2500* scans from -1 to +11 ppm *sw=2000* scans from 0 to +10 To scan from-0.5 to +8 you will need to adjust tof and sw. *sw=1750*, *tof=-250*	
4	To determine the how long your acquisition will take, type *time*.	
5	To change delay time, time *d1=x* where x is the new delay time. The default value is 1 sec. However, sometimes to obtain a good integration, d1 needs to be longer.	
6	For samples that are very concentrated in with protons such as samples intended for 13C analysis, you will often get an ADC overflow error. If this occurs, reduce the pulse width to 1 (type *pw=1*).	
7	To abort acquisition type *aa*.	9/11 jrs
8	To set up multiple experiments use the jexp command. Set up the first experiment in experimental window 1 by typing *jexp1* and start the experiment by typing *ga*. Then move to experimental window 2 by typing *jexp2*, set up and start, etc. Type *unlock(x)* where x is the value of the window if the experimental window is locked. See p. 16 for more details.	9/11 jrs
9	To allow experiment to access data in another experimental window, type *listenon*. When finished, type *listenoff*.	1/14/12

Processing NMR Spectra:

1) First phase the spectrum using the automatic phasing routine; type *aph*. (For advanced phasing: select the Process button on the lower menu bar. Select the Phase button. Click on a peak in the far left of the spectrum and phase it. Click on a peak on the far right and phase it.)

2) Select the Dscale button to display the scale.

3) Set the reference peak. Expand on the reference peak which is either TMS or the residual proton signal of the solvent. Left click on the peak to display the red cursor. Select the Ref button and follow instructions.
(Alternate method: place cursor on peak, type *nl*, type *rl*, enter reference value.)

4) Vertical scale adjustment: click on spectrum, hold center button on mouse and adjust up/down. (Alt: type *vs=x* where x is the new value.) Sometimes it is necessary to adjust the vertical position (*vp=x*).

5) Integrating the peaks in the spectrum:
In Interactive menu. Click on the integral button until Full Integral is displayed. Left click Reset before and after each peak that you want to integrate. If you make a mistake, right click to remove the reset. Click on the integral button until Part Integral is displayed. Place cursor on the peak that you want to use for setting the reference integral value. Click Set Int and input value.

To display integral values, type *dpirn*.
To print integral values, when you are in the plotting screen, type *pirn*.

6) Plotting the spectrum:
a) set the threshold by selecting Th and with middle cursor on mouse adjust the yellow line to capture the desired peaks.
b) select Display. In the following sequence, select Plot (to allow the spectrum to be plotted), Scale, Parameters, Peaks, type *pirn* (to display integral values) and Page. The last command is the print command. (Alt: select Autoplot.)

7) Save the spectrum:
a) Type *svf*.
b) Enter file name. If you are playing around, use the word "test" in the name so it can be easily deleted in the future.
c) **Directories have been created.** To save your file in a specific directory, click on File, select the desired directory, then click on Change then type *svf*. To return to the main directory, click on Home.

8) To retrieve saved files: Main menu, File, select file, Load. Note: you may need to change the directory.

Hints on Spectrum Processing

#	Item	Init/date
1	Convert from ppm to frequency: *axis='h'*	2/11 jrs
2	Convert from frequency to ppm: *axis='p'*	2/11 jrs
3	Autoplot will give a fully plotted spectrum with integration.	9/11 jrs
4	If the printer is offline, carry out the following sequence: Main Menu, more, Configure, Select Plotter.	9/11 jrs
5	To print shims (dgs) and 1H (or 13C) parameters (dg), *printon dg dgs printoff*	
6	To permanently modify 1H (and 13C) parameters: *rtp('/vnmr/stdpar/1Hpar')*, then make change, then *svp('/vnmr/stdpar/1Hpar')*.	
7	To check which directory you are in, type *pwd*.	1/14/12

Acquiring a $^{13}C\{^{1}H\}$ NMR Spectrum – Proton decoupled or ^{13}C(proton coupled)

Note: the ^{13}C(proton coupled) experiment has not been working correctly. Please run a DEPT to obtain CH attachments.

1) First lock and shim your sample and obtain a good ^{1}H NMR spectrum.
2) Select Setup, then select nucleus (^{13}C) and solvent.
3) For decoupled experiments, leave the decoupler set at *dm='nny'*. For ^{13}C(proton coupled) experiments use *dm='nnn'*.
4) The default number of transients is 1024 which requires ca. 30 min. If desired (for example, for an overnight experiment), change the number of transients (*nt=x,* where x is the number of transients, or *nt=-1* for infinite transients). To determine the length of the acquisition time, type *time.*
5) Select Acquire then Go, Wft. The status screen will appear and at the completion of the experiment, the NMR spectrum will appear.
6) You may periodically examine the spectrum by clicking on the Process menu button, Display FID, then fourier transform the FID. (Follow this procedure for *nt=-1*.)

You have now taken a ^{13}C NMR spectrum! Processing, displaying and printing is similar to ^{1}H NMR spectra. The *aph* routine works very well for ^{13}C spectra.

The experiment is not complete!

5) Turn off the decoupler! The decoupler used in this experiment generates heat and can also burn-out if left on for long periods of time. It is essential to turn it off. To do this, simply run a ^{1}H NMR spectrum.
6) To run sequential experiments use the *jexp* command. For example, set up the ^{13}C NMR experiment in experimental window 1, type *jexp 1*, then, start that experiment by typing *ga*. Move to experimental window 2 by typing *jexp2*, then, set up, for example, a ^{1}H experiment and start experiment by typing *ga*. You will notice that it is "QUEUED" in the Acquistion window.

Advanced ^{1}H and ^{13}C Experiments:

1) COSY
a. Run a ^{1}H NMR experiment and make sure the sample is locked and shimmed well.
b. Go to Main Menu, Setup, Sequence, 2D, COSY.
c. Adjust the number of transients (*nt=x*). If your sample gives a good quality ^{1}H NMR on 4 scans, you will be able to obtain a quality COSY is 20 min.
d. Type *time* to check the length of the experiment.
e. Type *su* to setup the experiment and *go* (not *ga*) to start acquisition.
f. There are two ways to process the experiment:

manual process: Type *wft2d* and wait until spectrum is processed (be patient as this takes a few minutes). Type *dconi* to allow for interactive scaling of the contour plot. Adjust level of contours as needed by selecting the color and intensity scale of the right y axis. Adjust the spectral window by clicking the cursor in the lower left corner of the desired part of the spectrum and drag with the right mouse button to the upper right corner. This will create a "box" outlined in red with the desired spectral range. To add the 1D spectra, click on Proj, then select horizontal projection (Hproj(max)), then click on Plot; then select vertical projection (Vproj(max)) then click on Plot. To print type *pcon page* or click on Autoplot.

autoprocess: Click on Autoprocess and be patient as the experiment takes a few minutes to process. To print, click on Autoplot.

Note: COSY files use a lot of memory, thus, do not save the file unless absolutely necessary.

2) DEPT (note pw90 must be properly determined, to check the value of pw90, type *pw90?*Type *dg* to display DEPT parameters and check to see that the pw value matches pw90.)
a. For this experiment, it is good to have a relatively concentrated sample > 50 mg/mL. Run a ^{13}C NMR experiment and note the minimum number of scans needed to obtain a spectrum with desired resolution.
b. Select Main Menu, Setup, Sequence, DEPT.
c. Set number of scans (nt=x). Note that the experiment performs 4 sets of scans at the set value of nt. Thus, these experiments can be quite long, especially, if the sample is dilute.
c. Type *su* to set-up the experiment and *ga* to start acquisition.
d. Processing: For manual processing type *adept*, *pdept*. Click on Autoplot to print. For autoprocessing, click on Autoprocess then Autoplot to print.
e. When this experiment is completed, please run a ^{1}H NMR to turn off the decoupler. Thank you!

Note: DEPT files use a lot of memory, thus, do not save the file unless absolutely necessary.

Performing Sequential Experiments

These instructions allow you to set-up several experiments at once and have them all run automatically! A sequence for ^{1}H, $^{13}C\{^{1}H\}$, and ^{1}H is described. This is a good sequence to run because it allows you to easily run a ^{1}H spectrum after a ^{13}C to turn off the decoupler.

1) Make sure you are in experiment window #1 by typing *jexp1*.**
2) Setup a ^{1}H NMR experiment and start the acquisition by selecting Go,Wft or type *ga*.
3) Enter experiment window #2 by typing *jexp2*.
4) Setup a $^{13}C\{^{1}H\}$ experiment. Be sure to select the appropriate number of transients. Type *time* to determine the length of the acquisition. Start the experiment by selecting Go, Wft or Go, Wft periodic. In the Acquisition Status Queued: 1 will be displayed indicating that when the experiment in window #1 is complete, the experiment in window #2 will start.
5) Type *jexp3*, then setup a ^{1}H NMR experiment and click on Go, Wft.
6) To view spectra, process, save and print, type *jexpx*, where x is the desired experiment window.

Note: it is best note to go too crazy with this and also to practice it a couple of times before doing a critical set of experiments.

**Occasionally an experiment window becomes locked and reads "foreground process is active" or window is active when it is not. To correct this, type *unlock(x)* where x is the experimental window you are trying to access (i.e., if *jexp1* is locked, type *unlock(1)*).

****Authorized users only****

Computer and Console Emergency Shutdown Procedure

Warning: the 200 NMR is designed to run permanently. Problems have occurred when attempting to turn the instrument back on. Thus, perform this procedure only when necessary (e.g., a scheduled power outage).

To turn off:

1) If you are running an experiment, type *aa* (to abort acquisition) then type *exit* to exit the vnmr program.
2) On the keyboard press the cresent moon key on the keyboard. Click Shutdown when instructed. This will turn off the computer.
3) ***Caution:*** **wait until the computer is fully turned off before proceeding to the next step.**
4) On the rear of the console, the main power switch is a white lever. Move this lever to the OFF position.
5) The computer and console is now off.

To turn on:

1) Turn on console by flipping main power switch to ON.
2) Turn on computer by pressing ON button on computer console.
3) Log into the vnmr program and test to see if computer and console are communicating. Frequently, these do not communicate. One remedy is to open the back of the console. While touching a piece of metal to ground yourself, wiggle the master clock circuit board. Another remedy is to repeat the turn off/turn on procedure until the computer and console communicate.

Liquid Nitrogen Fills

This is a very critical aspect of NMR maintenance. If the liquid nitrogen levels get too low, the liquid He will boil off rapidly and magnet will quench. This error costs many thousands of dollars to repair and months of valuable experimental time.

Checking liquid N_2 level: This must be done every Friday or Monday. Procedure: remove the cap labeled "Nitrogen Service" on the top of the magnet. Insert brown stick into the liquid nitrogen reservoir and count 30 seconds. This will cool the stick. Remove the stick and return the cap. Measure the frost level on the stick and write this into the notebook. It is considered an emergency if the N2 level is 2" or less.

Checking He and N_2 boil-off rate. This must be done once a day. Look on the left leg of the 200 magnet and locate the He and N_2 gauges. Record the level on the gauge as indicated by the floating metal ball. Write the levels into the notebook. Generally, the readings are 2 cc/h for He and 50 cc/h for N_2. If these are much higher, please contact Prof. Sowa or Prof. Murphy.

Liquid Nitrogen Fill:

Stop all acquisition experiments. Carefully roll the liquid nitrogen tank close to the 200 NMR. Attach ¾" rubber tubing to the "liquid" outlet on the tank. **Caution: the wrench you use for this is magnetic and may suddenly be attracted by the magnet. Please avoid this.** Attach the end of the tubing to the specially designed "T" metal tube and insert metal tube into the "Nitrogen Service" port. Slowly open the valve on the nitrogen tank and carefully increase flow until you can hear liquid nitrogen being dispensed. Maintain flow at reasonable rate until the nitrogen dewar in the magnet is filled, then, close the valve on the tank. Allow the rubber tubing to warm so that the rubber is flexible (you may need to carefully warm the tubing with a heat gun.) Detach the metal tube and place it back on the storage rack.

NMR Training Program

Contact Prof. Sowa to obtain training.

Complete the following exercises and submit work to Prof. Sowa for evaluation. Please print and scan each spectrum and submit report electronically.

1) Lock and shim on Dummy sample. Report Z0, lockpower, lockgain, lockphase, Z1c, Z2c and chemical shift. Print and scan spectrum.
2) Do either a or b.
    a. Prepare a sample of glucose (30 mg in 0.75 mL D_2O). Lock and shim on sample. Take 1H NMR. Report chemical shifts and any coupling constants that can be resolved. Integrate the spectrum and obtain integrations within 20 % of the expected value. $^{13}C\{1H\}$ nmr (proton decoupled) and report chemical shifts (note; for the 200, ppm values are good to 2 decimal points). Correlate chemical shifts with structure. Print and scan spectrum.
    b. Follow above procedure except using menthol dissolved in $CDCl_3$ or C_6D_6.
3) Lock and shim on $CDCl_3$. Report Z0, lockpower, lockgain, lockphase, Z1c, Z2c values that you found. Take $^{13}C\{^1H\}$ and report chemical shift and $J^{13}C\text{-}^2H$ values.
4) Return Dummy sample, lock, shim and run ^1H.

References

These excellent books for performing NMR experiments are available in the SHU Library:

1. *Modern NMR Techniques for Chemistry Research*, Andrew E. Derome, Pergamon Press: New York, 1987.

2. *200 and more NMR experiments : a practical course*, Stefan Berger, Siegmar Braun, Wiley-VCH: Weinheim, 2004.

3. *Modern NMR spectroscopy : a guide for chemists*, Jeremy K.M. Sanders, Brian K. Hunter, Oxford University Press: New York, 1993.

These are excellent on-line references:

1. G. A. Pearson on How to Shim an NMR Magnet (http://nmr.chem.uiowa.edu/manuals/Shimming-GAP-NMR-magnet.pdf, accessed 9/30/2011).
2. Spectral Database for Organic Compounds, http://riodb01.ibase.aist.go.jp/sdbs/cgi-bin/direct_frame_top.cgi, accessed 9/30/2011.
3. University of Indiana NMR Facility, Training and Guides (see links): http://nmr.chem.indiana.edu/nmrblog/?page_id=2, accessed 9/30/11.

Useful articles:

1. "NMR Chemical Shifts of Common Laboratory Solvents as Trace Impurities," Hugo E. Gottlieb, Vadim Kotlyar, Abraham Nudelman, *J. Org. Chem.* **1997,** *62,* 7512-7515.

2. "NMR Chemical Shifts of Trace Impurities: Common Laboratory Solvents, Organics, and Gases in Deuterated Solvents Relevant to the Organometallic Chemist," Gregory R. Fulmer, Alexander J. M. Miller, Nathaniel H. Sherden, Hugo E. Gottlieb, Abraham Nudelman, Brian M. Stoltz, John E. Bercaw, Karen I. Goldberg, *Organometallics* **2010**, *29*, 2176–2179.

http://dx.doi.org.10.1021/om100106e

NMR Duties

General NMR Maintenance:

1. Room maintenance (printers, sweep floor, log books, book shelves, white board)
2. Left over NMR samples….
3. Cryogen flow meter readings (N_2(l) and He(l))
4. Cryogen level readings (200: N_2 level; 500 N_2, He level)
5. Liquid nitrogen fills
6. 200 Spectrometer Calibration
7. 500 Spectrometer Calibration (Update shim maps, Line shape test , Tune ^{1}H and ^{13}C frequencies)

8. Other_____

NMR Contributions Wish List

9. Contribute an update or correction to the NMR SOP manuals.
10. Export data from 200.
11. Set-up routines for APT, DEPT, INEPT, COSY, HETCOR for 200.
12. For 500: develop routines for multinuclear NMR (2H, 19F, 31P, …).
13. For 500: develop advanced 2-D, 3-D experiments.

14. Other_____

NMR Training: 200 NMR

15. Introduction to NMR Spectroscopy (theory and instrument)
16. SafetyThursday, September 15, 2011
17. Maintenance requirements
18. Sample preparation
19. Locking deuterium signal
20. Optimizing shimming
21. Optimizing lockphase
22. Set-up and acquire 1D NMR ^{1}H and ^{13}C spectra
23. Process 1D NMR
    (phasing, referencing, integrations, peak picking)
24. Saving data
25. Return dummy sample, lock, shim and take 1H spectrum
26. Evaluation: Perform the following a given set of experiments, give hard copy of print-out or scan and email report to Prof. Sowa.

Once you have completed training on the 200 and demonstrated expertise by completing the above evaluation, you may apply for training on the 500.