

Bruker Avance NMR Spectrometers
UIC Chemistry / RRC-East NMR Lab

Training Guide for Basic 1D NMR Spectroscopy

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Updated 02-01-01: updates reflecting the addition of the BBO probe for the Avance-400; JSH.

Updated 02-01-00: updated the parameter set listing, various typos; JSH.

Updated 10-27-99: Added discussion of the view command (pg. 5), updated the command list (pg. 7) and parameter set list (pg. 8), added the homonuclear decoupling discussion (pg. 12) and this Table of Contents.

**Basic Spectrometer Operation Guidelines -
Bruker Avance NMR Spectrometers
UIC Chemistry / RRC-East NMR Lab**

Logging on to the SGI O2 computer and starting XWINNMR

1. Click on the icon with your login ID or enter your login ID in the login field
2. Enter your password in the password field.
3. After the window manager starts, you will see two windows on the screen: one is the "console" window and one is a UNIX shell window. Move the mouse pointer to the UNIX shell window and type `xwinnmr` to start the NMR program.
4. The NMR program will start and you will see the main window appear.

Changing samples and shimming

5. Move the mouse cursor to the main window and type `lockdisp` to display the lock window. A sample of CDCl_3 resides in the magnet when the spectrometer is not in use, and this sample will be spinning and will be locked.
6. On the keypad, turn off LOCK and SPIN functions, then press the LIFT key to eject the CDCl_3 standard sample. NOTE: the lift function on the Avance spectrometers does not require the orange shift key. NOTE: DO NOT TOUCH THE MAGNETS!! They are on pneumatic legs and will move!
7. After placing your sample (CLEAN IT!!) in the spinner and inserting the spinner in the magnet, press the LIFT key again to insert the sample and press the SPIN button to start spinning.
8. Make sure the main window is highlighted and type `lock`. A new window will appear with a list of solvents; click the appropriate solvent using the mouse pointer. Wait for lock to be established.
9. Shim your sample using Z1, Z2 and Z3 shims as on the older Bruker's. Adjust the LOCK GAIN and/or LOCK POWER as necessary. Shimming is a little more demanding on the Avance machines than on the older Bruker's so be prepared to take your time. NOTE: on the DRX-500 keypad, the ON AXIS function must be set on prior to shimming.

10. After shimming is completed you may either close (iconify) the lock window by clicking the small "dot" button in the upper right border or you can leave it open behind the other windows. Click on the spectrum window to make it active.

Data acquisition and Fourier transformation

11. If you do not want to use the currently displayed dataset for your acquisition, type `edc` (edit current dataset) to create a new one. When the `edc` window appears, type in the new dataset name in the NAME field and type in the desired experiment number in the EXPNO field (the PROCNO should be 1 and the DISK (/vsr), USER (your login ID) and TYPE (nmr) fields should not be changed) then click on SAVE to close the window. NOTE: the EXPNO can be any number but typically I use 1 for the proton spectrum, 2 for carbon, 3 for dept, 4 for COSY, etc. NOTE: the `edc` command can also be used to read existing datasets.

12. Type `rpar h1.bbo` to read the standard proton parameters, `rpar c13.bbo` for the standard carbon-13 parameters, etc. A window will appear listing the different components of the parameter set; click the COPY ALL button to load all relevant parameters (OR, click the parameter types you want to load (they will then be highlighted) and then click COPY). All of the standard parameter sets are listed on pg. 7 of this handout. NOTE: on both spectrometers there are two probes available and the .xxx extension on the parameter file must correspond to the correct probe. There is a message in the UNIX windows telling you which probe is in use.

13. To examine the acquisition parameters you can use either the `eda` (edit acquisition parameters) or the `ased` (acquisition setup editor) commands. Each brings up a window; after examining and changing anything (if necessary), click the SAVE button to close the window and save the changes, or click the CANCEL button to close the window without saving any changes.

14. Type `acqu` to display the acquisition window (for FID display).

15. Type `rga` to set receiver gain - wait for message telling you it is finished.

16. Type `zg` to start acquisition. The command `halt` can be used to stop an acquisition before the requested number of scans (`ns`) is completed. NOTE: during a longer acquisition (e.g. C13) the command `tr` can be used to transfer the FID to disk. The `ef` command will then transform that FID and display the result. Use the `acqu` command again to return to the FID display.

17. After acquisition is finished type `ef` to do the Fourier Transform. NOTE: after transformation the spectrometer will automatically switch to the spectrum display window.

18. To examine the processing parameters you can use the `edp` (edit processing parameters) command. This brings up a window; after examining and changing anything (if necessary), click the SAVE button to close the window and save the changes, or click the CANCEL button to close the window without saving any changes. NOTE: the FID is always stored on disk so the `ef` command can be retyped at any time to generate a spectrum, e.g., after a processing parameter has been changed.

Data processing and plotting

19. **PHASING:** Using the left mouse button, click the PHASE button on the menu to the left of the spectrum display to enter the phasing routine. Click the BIGGEST button to apply a default phase correction using the biggest peak in the spectrum as the reference point. Then, move the mouse pointer over the PH0 button, hold down the left mouse button and move the mouse vertically up and down to adjust the 0-order phase correction. When the biggest peak is phased correctly, repeat the above process using the PH1 button, then iterate back and forth between PH0 and PH1 until the entire spectrum is phased correctly.

20. Click on the RETURN button to exit phasing; click the SAVE & RETURN choice that appears to save your phase corrections.

21. **SPECTRUM MANIPULATION:** To expand the spectrum and to move and/or change the expanded region, use the arrow buttons to the left of the spectrum display. These buttons have labels such as `<`, `>`, etc. In addition, if the mouse is moved over the spectrum window and the left button clicked, the mouse pointer will jump onto the spectrum to give the chemical shift readout. The middle mouse button will then freeze the cursor in the desired location. Moving the mouse again will move a second cursor, and clicking the middle button again will expand the region between the cursors. Now click the left mouse button to release the cursor from the spectrum.

Other manipulations:

- *2, /2, *8, /8: click these buttons to adjust vertical scale up or down by factor 2 or 8;
- move the cursor over the spectrum, hold the middle mouse button down: move mouse left - right to scroll spectrum left - right;
- move the cursor over the spectrum, hold the right mouse button down: move mouse left - right to expand or contract spectrum.

22. **DEFINE PLOT REGION:** To define a region for plotting, click on the DP1 button to the left of the spectrum display. A window will appear asking for the plot limits. If you want to plot the spectrum region as displayed, just press return twice; if you want to plot a specific region, enter the desired downfield and upfield plot limits and then y to the scale question, and that region will then be displayed.

23. If you want to expand the spectrum vertically for plotting, type cy and enter the desired value; the default is 12 - 15 cm.

24. **TITLE:** To enter a title for the plot, type setti. A text-editor window will open. This window uses the IRIX Jot graphical editor. The default title will be shown. Move the mouse pointer to the Jot window and edit the title as you would using any graphical text editor. When you are finished, move the mouse pointer to the FILE button in the upper right of the window. Hold down the left mouse button and move the pointer down the pop-up menu to the SAVE choice and then release the button; this will save the text file. Repeat this process, and go to the EXIT choice to close the Jot window.

25. **PEAK PICKING:** To check the peak picking, type pps. This will bring up a new window with the peaks listed. Click on the OK button to close this window. If you want to change the peak picking threshold, type mi and enter a new value in the window that appears. Repeat pps if necessary.

26. **INTEGRATION:** Click on the INTEGRATE button to the left of the spectrum window. Move the mouse pointer to the spectrum and click the left mouse button to put the cursor on the spectrum. Move the cursor to the downfield limit of integration and click the middle mouse button; move the cursor to the upfield limit and click the middle button again. Move the cursor back downfield a little and click the left mouse button to highlight this new integral trace (the trace will then show an asterisk at its end). Now move the pointer over to the left-side buttons and place

the pointer over the BIAS button. Hold down the left mouse button and move the mouse up and down to adjust the bias (similar to how the phasing was done); repeat the process using the SLOPE button until the integral trace looks correct. NOTE: the bias effects the whole integral trace equally while the slope is frequency dependent with the downfield end of the trace as a reference point.

27. Move the pointer back to the spectrum and click to left mouse button to place the pointer on the spectrum. Now move the cursor on the spectrum to the most downfield break point desired in the integral trace. Click the middle mouse button to break the integral trace, move the mouse upfield the desired amount, and click the middle mouse button again to restart the integral trace. Repeat as necessary. When all the desired regions are defined, highlight one of a known area with the left mouse button, move the pointer to the left of the spectrum and click on the CALIBRATE button, then enter the desired integral area in the window that appears.

28. When the integration is complete, adjust the vertical scale of the integrals using the *2 or /2 buttons adjacent to the ALL button, then click on the RETURN button to exit; click the SAVE & RETURN choice that appears to save your integrals.

29. PLOTTING:

a) Prior to plotting the spectrum on paper, type `view` to see the plot output displayed on the screen (in a new window). Check that the plot is correct prior to plotting to paper. Click on the QUIT button in this new window to close the view window.

b) To plot the spectrum on paper using the default parameters, type `plot`. This will plot the spectrum, the axis, the integration (proton only), the peak picking, the title, and the parameters. If you want to alter this, type `edg` (edit graphics parameters). A window will appear with buttons for the choices regarding the items included in the plot; after examining and changing anything, click the SAVE button to close the window and save the changes, or click the CANCEL button to close the window without saving any changes. If you delete integration and/or peak picking, increase `cy` prior to issuing the plot command.

Finishing up with XWINNMR and logging off the SGI O2 computer

30. Click on the LOCK DISP icon or on the border of the lock window to open the window or bring it to the front.

31. Remove your sample and replace it with the CDCl_3 standard following steps 6 & 7 above.

32. Type `rsh shims.bbo` to load the standard CDCl_3 shims. NOTE: on both spectrometers there are two probes available and the `.xxx` extension on the shim file must correspond to the correct probe. There is a message in the UNIX windows telling you which probe is in use.
33. Lock and shim the CDCl_3 standard sample following steps 8 & 9 above.
34. Click the QUIT button in the lower right corner of the lock window to close the lock window.
35. If you have been running spectra other than proton, use the `edc` command (step 11) to read a proton dataset, and then type `ii` to initialize the spectrometer back to proton.
36. Type `exit` to leave the NMR program. A confirmation window will pop up - READ IT! If it asks "Do you want to leave XWINNMR?", click the OK button. If it says "Processes are still active - Kill them?", click the CANCEL button. Go back to the XWINNMR window and double check that the lock window is not still running and hidden somewhere. Type `lockdisp` to re-display the lock window, then click the QUIT button in the lower right corner of the lock window to close the lock window. Type `exit` again - if processes are still active, call NMR Facility staff for assistance.
37. Move the mouse pointer to the background of the UNIX desktop and hold down the right mouse button. A desktop menu will appear. Keep the right mouse button depressed and move the cursor to the LOG OUT choice, then release the right mouse button. A logout screen will appear: click YES with the left mouse button to logout from UNIX.

Notes

XWINNMR

Commonly Used Keyboard Commands

NOTE THAT ALMOST ALL COMMANDS ARE AVAILABLE FROM THE PULL-DOWN MENUS AT THE TOP OF THE SPECTRUM DISPLAY

Parameter Setup

edc - edit current dataset - reads existing and/or creates new datasets
 eda - edit acquisition parameters
 ased, edasp - edit acquisition parameters - pulse program driven or nucleus-related
 edte - set up temperature controller parameters
 edp - edit processing parameters
 edg - edit graphics parameters - controls plot output attributes
 wrpa - copy current dataset to a new one - specify new name and experiment number
 re - read new dataset - specify new name and experiment number

Data Acquisition

lockdisp - display lock window
 lock - start autolocking routine
 rpar - read parameter set
 rsh - read shim set
 ii - initialize interfaces
 acqu - display acquisition window - shows FID on screen
 wobbb - start wobble routine for probe tuning
 rga - set receiver gain automatically
 ns - number of scans
 zg - zero current data and start acquisition
 go - start data acquisition
 tr - transfer FID to disk for processing
 halt - halt data acquisition after next scan
 stop - stop data acquisition immediately

Data Processing and Plotting

setti - enter title for plot
 ft - Fourier transformation
 ef - exponential multiplication and Fourier transformation
 em - exponential multiplication
 lb - controls amount of exponential multiplication
 nzp - number of data points to zero at start of FID
 zp - zero nzp points at start of FID
 basl - enter baseline correction routine
 pscal - define plot vertical scaling method
 cy - plot vertical scaling
 cx - plot horizontal scaling
 mi - threshold for peak picking
 pps - peak picking with output on screen
 pp - peak picking with output on paper
 view - view plot output on screen
 plot - plot spectrum on paper
 dual - enter dual display mode - EXPNO2 and PROCNO2 parameters must be correct in the menu

Avance Spectrometer Parameter Files - 02/01/01

DPX-400 (BBO probe)

h1.bbo
homodec.bbo
f19.bbo
c13.bbo
c13dept.bbo
p31.bbo
p31nd.bbo
cosy.bbo
hmqc.bbo
hmbc.bbo
tocsy.bbo
noesy.bbo
shims.bbo

DPX400 (BBI probe)

h1.bbi
homodec.bbi
c13.bbi
c13dept.bbi
p31.bbi
p31nd.bbi
pt195.bbi
se77.bbi
si29.bbi
cosy.bbi
hmqc.bbi
hmbc.bbi
tocsy.bbi
noesy.bbi
shims.bbi

DRX-500 (BBO probe)

h1.bbo
homodec.bbo
f19.bbo
c13.bbo
c13dept.bbo
p31.bbo
p31nd.bbo
b11.bbo
h2.bbo
o17.bbo
pt195.bbo
si29.bbo
cosy.bbo
hmqc.bbo
hmbc.bbo
tocsy.bbo
noesy.bbo
shims.bbo

DRX-500 (TBI probe)

h1.tbi
homodec.tbi
c13.tbi
c13dept.tbi
p31.tbi
p31nd.tbi
cosy.tbi
hmqc.tbi
hmbc.tbi
tocsy.tbi
noesy.tbi
shims.tbi
shimsn2ns.tbi (for water suppression using N2 VT gas)

Probe Tuning Guidelines - BBI, TBI and BBO Probes
Bruker Avance NMR Spectrometers
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Spectrometer setup - H1 tuning

1. Run a quick proton experiment to determine that the sample is correct.
2. We will tune the H1 coil first. Turn off the sample spinning.
3. Type `acqu` to go to the acquisition window, then type `wobb` to start the probe tuning ("wobble") routine.

Tuning the H1 coil

4. The computer screen will show a trace with a dip in it; hopefully the dip will be close to the correct frequency in the center of the screen. If there is no dip, click the `WOBB-SW` button to the left of the screen, then enter a new value in the correct field of the screen that pops up. The default SW is 4 MHz.; try 12 or 16 MHz. if it needs changing.
5. When you have confirmed that there is a dip present, go to the magnet and adjust the tune and match screws under the probe to bring the minimum of the dip to the center of the screen and also as close to the bottom of the display as possible. NOTE: the location of the dip left to right is referred to as the "tune" and the depth of the dip (closeness to the baseline) is referred to as the "match." The tune screw will tend to move the dip left - right and the match screw will improve the depth of the dip. The display on the preamp housing also shows the quality of the probe tuning and can be used in conjunction with or instead of the computer screen display. NOTE: on the TBI probe on the DRX-500 the proton coil tune and match is done using the small screwdriver-like rods and the special tool hanging off the probe.
6. When the tuning is OK, type `halt` to stop the wobble routine.
7. Restart sample spinning if desired. NOTE: leave spinning off for 2D experiments.
8. Re-acquire the proton spectrum if desired.

Spectrometer setup - X-nucleus tuning

1. Carry out steps 1 - 8 above to ensure the H1 coil is tuned.

2. Use the `edc` command to read or create a new dataset.
3. Use the `rpar` command to read the correct parameter set for the X-nucleus you want to observe, then type `ii` to initialize the spectrometer. Turn off sample spinning.
4. Type `acqu` to go to the acquisition window, then type `wobb` to start the probe tuning ("wobble") routine.

Tuning the X-nucleus coil

5. X-nucleus coil tuning is done using gold-colored sliders visible on the bottom of the probe. The sliders are numbered, and there is a directory of numbers for the tune and match settings for most common X-nuclei hanging below the probe. Confirm that the tune and match settings are correct for the X-nucleus you want to observe. NOTE: on the TBI probe on the DRX-500, the X-BB coil is not used for C13 - there is a dedicated C13 coil for that. The C13 coil is tuned using the single blue screw. The X-BB coil is tuned in the same fashion as the other probes. NOTE: For observing X nuclei other than C13 with the TBI probe, the cable from the middle (vertical) connection on the preamplifier has to be connected to the X-BB coil connection on the probe.
6. The computer screen will show a trace with a dip in it; hopefully the dip will be close to the correct frequency in the center of the screen. If there is no dip, click the WOBB-SW button to the left of the screen, then enter a new value in the correct field of the screen that pops up. The default SW is 4 MHz.; try 12 or 16 MHz. if it needs changing.
7. When you have confirmed that there is a dip present, go to the magnet and adjust the gold tune and match sliders under the probe to bring the minimum of the dip to the center of the screen and also as close to the bottom of the display as possible. NOTE: the location of the dip left to right is referred to as the "tune" and the depth of the dip (closeness to the baseline) is referred to as the "match." The tune slider(s) will tend to move the dip left - right and the match slider(s) will improve the depth of the dip. The display on the preamp housing also shows the quality of the probe tuning and can be used in conjunction with or instead of the computer screen display.
8. When the tuning is OK, type `halt` to stop the wobble routine.
9. Restart sample spinning if desired. NOTE: leave spinning off for 2D experiments.
10. Be sure to tune the X-nucleus coil back to C13 on the BBI and BBO probes before you leave the spectrometer. The X-BB coil on the TBI probe is tuned to P31 by default.

Avance Spectrometers - Probe Tuning Matrix

Which experiments are likely to require probe tuning?

TYPE OF EXPERIMENT:	H/F NUCLEI:		X NUCLEI:		
	<u>1H</u>	<u>19F*</u>	<u>13C</u>	<u>31P</u>	<u>OTHER</u>
1D obs. no dec.	No	Yes	Maybe	Yes	Yes
1H homonuc. dec.	Yes	No	No	No	No
1D X obs. 1H dec.	Maybe	Yes	Maybe	Yes	Yes
1D X obs. DEPT	Yes	Yes	Yes	Yes	Yes
1D 1H obs. X dec.	Yes	Yes	Yes	Yes	Yes
2D Simple COSY	No	Yes	N/A	N/A	N/A
2D 1H-only other	Yes	Yes	N/A	N/A	N/A
2D 1H obs. X dec.	Yes	Yes	Yes	Yes	Yes
2D X obs. 1H dec.	Yes	Yes	Yes	Yes	Yes
3D, 4D any	Yes	Yes	Yes	Yes	Yes

*¹⁹F only possible with the BBO probes. ¹⁹F - ¹H experiments are not possible with our existing probes.

Instrumental Overview - Sensitivity (S/N), 5mm Probes

<u>Console/Probe</u>	<u>¹H sensitivity</u>	<u>¹³C sensitivity</u>
AM-400/Nalorac BB	160:1	130:1
Avance DPX-400/BBO-Z-grad	220:1	160:1
Avance DPX-400/BBI-Z-grad	650:1	110:1
Avance DRX-500/BBO-Z-grad	330:1	220:1
Avance DRX-500/TBI-Z-grad	750:1	90:1

As can be seen from the chart, the indirect-detection probes on both of the new instruments provide huge increases in sensitivity for ¹H observation. The BBI-Z-gradient (broadband-inverse) probe on the DPX-400 provides roughly the same ¹³C sensitivity as does the Nalorac BB probe on our old AM-400, even though the BBI-Z-gradient probe is not optimized for X-nucleus observation. The same is true of the TBI-Z-gradient (triple-resonance broadband-inverse ¹H-¹³C-X) probe on the DRX-500, except this probe suffers for ¹³C observation because the ¹³C coil is double-tuned and optimized for ¹H observation. The BBO (broadband-observe) probes offer a significant increase in ¹³C and other X nuclei sensitivity for direct X-nucleus observation. As of January 2001, the BBO probes will be the default probes for both spectrometers.

**Proton Homonuclear Decoupling Instructions -
Bruker Avance NMR Spectrometers
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1. Run the conventional proton spectrum using the standard parameters as described above.
2. Process and phase the proton spectrum as normal.
3. Type `wrpa` followed by a new experiment number to copy the current dataset to a new dataset with the requested experiment number. For example, if the current dataset has the name "smellythiol" and the experiment number "1", typing `wrpa 2` will copy "smellythiol / 1" to "smellythiol / 2".
4. Type `re` followed by the new experiment number to go to the new dataset. Following the example above, type `re 2` to go to experiment number 2.
5. In the new dataset, type e.g. `rpar homodec.bbo all` to read all the parameters for the homodecoupling experiment.
6. Check the tuning of the proton coil as described elsewhere.
7. **SET THE DECOUPLER FREQUENCY:** Expand the spectrum about the peak you want to decouple. Click with the left mouse button on the UTILITIES button to the left of the spectrum. Then click on the O2 button with the left mouse button. Then move the mouse pointer to the spectrum area (the pointer will jump to the spectrum) and place the cursor on the center of the multiplet you want to decouple. Click the middle mouse button to define this frequency as the decoupler frequency (parameter O2). Then click the RETURN button on the lower left of the screen to return to the normal spectrum display.
8. Type `RGA` to set the receiver gain. After this completes type `ZG` to run the decoupled spectrum. Process in the normal fashion.
9. Expand the decoupled peak to check for complete decoupling. If decoupling is not complete, adjust the decoupler power. To do this, type `PL14` and enter a new value that is 3 units lower numerically than the current value (i.e., if the current value is 45, enter 42). Do not enter a PL14 value less (numerically lower) than 30! Repeat the experiment to check decoupling.

10. COMPARING SPECTRA USING THE DUAL DISPLAY MODE: To compare the decoupled and undecoupled spectra, type `dual` to enter the dual display mode. A menu will pop up containing fields to enter the information needed to define the second data set. Click the `EXPNO2` button and enter the experiment number of the reference (undecoupled) spectrum - probably this will be 1. Click the `PROCNO2` button and enter 1. Then click the `DONE` button on the lower left of the pop-up window, and the spectrum display will now have the decoupled spectrum in green and the reference (undecoupled) spectrum in purple displayed together. The buttons on the upper left can be used to expand and manipulate the display. Click the `RETURN` button on the lower left when you are finished with the dual display mode.

11. Repeat steps 7 and 8, above, to decouple other peaks as needed.

**Laboratory Use Rules -
Bruker Avance NMR Spectrometers
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Care of Magnet and Spectrometer Hardware

1. Clean your NMR tubes before inserting into spinner and before putting in magnet.
2. Only good-quality NMR tubes are permitted.
3. **DON'T TOUCH THE MAGNETS!** They are on pneumatic vibration-dampers and will move!
4. If you are training someone, you must accompany the trainee at all times.

UNIX Computer Use

1. Overall message - these computers are for NMR data acquisition, processing and related research **ONLY!**
2. **NO net surfing!** - netscape and other web-related directories will be removed without warning!
3. **NO downloading of executable codes!** - unrecognized files will be removed without warning!
4. **NO sending or receiving of email to or from outside chemistry!**
5. **DO NOT** give your account password to any other person.
6. Keep disk-space quotas in mind.
7. **ARCHIVE YOUR DATA** either with a Jaz disk or via ftp. Facility staff **DO NOT** archive data.
8. Check your normal email address frequently as I will email information about the labs.
9. Currently, the computers are set up with NFS-mounted user home directories. This means that a user **CANNOT RUN BOTH** spectrometers at the same time.

RRC and Laboratory - Related Issues

1. This new lab and equipment is a showcase for our campus - keep it clean and neat!
2. Do not give your ID card, or the codes for the door locks, to any other person.
3. If you use the wetlab, keep it clean also. Label clearly any materials you are leaving there.
4. Cryogen fills will be usually Tuesday, Wednesday, or Thursday, midday.
5. Keep in mind that problem users will have their access terminated.

**Group Expert Checkout Requirements -
Bruker Avance NMR Spectrometers
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This is an outline of the tasks you will be required to perform correctly in order to pass the checkout exam. You will be allowed up to one hour to complete the following tasks. You may use any training handouts and notes you have. Standard samples will be used for the checkout and will be provided by the NMR lab. The checkouts will be done using the DPX-400 with either the BBO or BBI probe. Dr. Harwood will observe you performing the checkout at his discretion.

Sample 1 - 10% Ethylbenzene in CDCl₃

- login to computer and start XWINNMR, open lock display
- remove CDCl₃ standard, insert new sample, lock and shim
- create new dataset
- run ¹H spectrum, process, integrate and plot
- set up new dataset for ¹³C spectrum
- check probe tuning for ¹H and ¹³C
- run ¹³C spectrum, process and plot

Sample 2 - 0.0485 M TPP in CDCl₃

- remove sample 1 and replace with sample 2, lock and shim
- go back to ¹H dataset, run ¹H spectrum
- set up new dataset for ³¹P spectrum
- tune probe for ³¹P
- run ³¹P spectrum, process and plot
- retune probe to ¹³C

Standard Sample - 100% CDCl₃

- remove sample 2 and replace with CDCl₃ standard sample, lock and shim
- initialize spectrometer to ¹H
- shutdown XWINNMR and logout from UNIX