



200 MHz NMR Spectrometer

Training Guide for Basic 1D NMR Spectroscopy

Mercury 200 with 5mm 4-nucleus probe

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SAFETY PRECAUTIONS

The following warning and caution notices illustrate the style used in Varian manuals for safety precaution notices and explain when each type is used:

WARNING: *Warnings are used when failure to observe instructions or precautions could result in injury or death to humans or animals, or significant property damage.*

CAUTION: *Cautions are used when failure to observe instructions could result in serious damage to equipment or loss of data.*

Warning Notices

Observe the following precautions during installation, operation, maintenance, and repair of the instrument. Failure to comply with these warnings, or with specific warnings elsewhere in Varian manuals, violates safety standards of design, manufacturing, and intended use of the instrument. Varian assumes no liability for customer failure to comply with these precautions.

WARNING: **Persons with implanted or attached medical devices such as pacemakers and prosthetic parts must remain outside the 5-gauss perimeter from the centerline of the magnet.**

The superconducting magnet system generates strong magnetic fields that can affect operation of some cardiac pacemakers or harm implanted or attached devices such as prosthetic parts and metal blood vessel clips and clamps.

Pacemaker wearers should consult the user manual provided by the pacemaker manufacturer or contact the pacemaker manufacturer to determine the effect on a specific pacemaker. Pacemaker wearers should also always notify their physician and discuss the health risks of being in proximity to magnetic fields. Wearers of metal prosthetics and implants should contact their physician to determine if a danger exists.

Refer to the manuals supplied with the magnet for the size of a typical 5-gauss stray field. This gauss level should be checked after the magnet is installed.

WARNING: **Keep metal objects outside the 10-gauss perimeter from the centerline of the magnet.**

The strong magnetic field surrounding the magnet attracts objects containing steel, iron, or other ferromagnetic materials, which includes most ordinary tools, electronic equipment, compressed gas cylinders, steel chairs, and steel carts. Unless restrained, such objects can suddenly fly towards the magnet, causing possible personal injury and extensive damage to the probe, dewar, and superconducting solenoid. The greater the mass of the object, the more the magnet attracts the object.

Only nonferromagnetic materials—plastics, aluminum, wood, nonmagnetic stainless steel, etc.—should be used in the area around the magnet. If an object is stuck to the magnet surface and cannot easily be removed by hand, contact Varian service for assistance.

Basic Spectrometer Operation Guidelines – 1D Spectra
Varian 200 MHz NMR Spectrometer
IPFW

Introduction

a. Magnet

The magnetic field is generated by a coil of superconducting wire immersed in a Dewar of liquid helium. This is surrounded by a vacuum chamber which is in turn surrounded by a Dewar of liquid nitrogen. The central bore contains shim coils that are used to adjust the field homogeneity in the vicinity of the sample.

Our magnet is an Oxford 200 MHz Narrow Bore type. Care should always be taken especially when close to the magnet (within the 5 gauss line). See the precautions signage.

b. Probes

The NMR sample is placed in a probe inside the magnet and arranged so that sample sits in the center of the magnetic field. The probe contains a set of coils that generate the Electromagnetic Field of the Radiofrequency (RF) pulses and also detect the signal induced by the effects of the pulse(s) on the sample. In addition the probe contains a heating element for temperature stability and also a gradient coil for applying magnetic field gradients.

c. Lock Circuit

A fixed deuterium frequency is generated by a frequency synthesizer. The signal is passed through a transmitter which controls the input of the deuterium signal into the probe. The signal is also amplified before going to the probe. Inside the magnet assembly a lock circuit to feed back loop mechanism. This unit detects changes in the deuterium frequency and reports these so as to adjust the magnetic field to compensate for these changes by changing currents to a shim coil.

d. Transmitter

The frequency of the observe channel is generated by a frequency synthesizer. The timing of the pulses in NMR experiments is controlled by a pulse programmer that acts in concert with the transmitter.

e. Detector

The signal from the observe coil is amplified by a preamplifier to increase the signal. The latter is then combined with a second frequency generated by a frequency synthesizer in a MIXER. The result is a 10.5 MHz signal for the 200 MHz spectrometer that is split into two phase sensitive detectors (PSD). These are 90° out of phase with each other. These detectors rectify the NMR signal with the intermediate frequency from the frequency synthesizer to give a signal in the audio frequency range (kHz). The latter is passed into the A/D converter to convert to a digital form that can then be stored and processed by a computer.

f. Decoupler

One decoupler circuit is used to simplify heteronuclear NMR spectra by removing the effects of ^1H -X nucleus coupling. This is done using a frequency synthesizer through a transmitter which is again controlled by a pulse programmer.

g. Gradient Amplifier

Most modern NMR spectrometers have gradients. These units permit much more efficient collection of 2D NMR since phase cycling is not required. These are acoustic amplifiers that apply a magnetic field across the sample. These units are controlled by the pulse programmer.

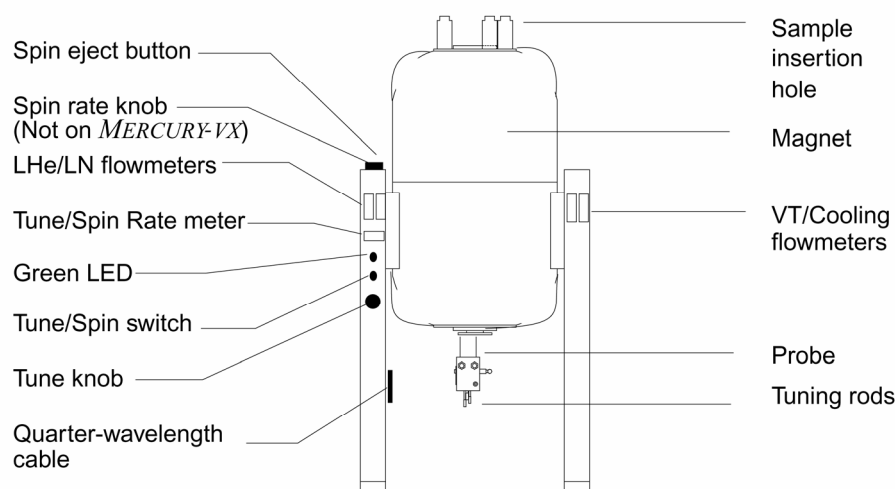


Figure 1. Magnet, Probe and Associated Electronics

Please note the following general policies

- Under no circumstances lean against or otherwise move the magnet
- Each NMR users is given an account (ID and password) on the spectrometer. Users in the same group share the same vnmrsys directory and so one must be careful that your actions do not disrupt others.
- Varian VNMR software enables you to easily access and change functions, options, and their properties. If you are not sure about an option, button or command, contact the IPFW NMR facility personnel for assistance.
- If you encounter problems at any time during locking, shimming, or acquiring data you should halt the experiment and reload standard parameters for your experiment
- In order for shimming to work well, samples should be filtered and prepared in good quality NMR tubes. Sample volumes should be at least 0.7 mL. Do not use more than 0.1% TMS.
- There are two devices to use to insert samples in the spinner turbines. The plastic gauge requires that the NMR tube is inserted until reaches the bottom.

- You must soak the NMR tube with hexane to lubricate before inserting. Also wipe (use Kimwipes) the tube with hexane and any fingerprints from the turbine to clean. Re-check the level with the gold metal gauge to be certain that the correct sample depth is used. Hold the turbine body, not its top, to keep from transferring fingerprints to the stack.
- The VNMR software works best with the Common Desktop Environment (CDE) graphics on the Sun computer. Please check when you login that you are using CDE.

Conventions in this guide

- Buttons are indicated with small caps in bold, e.g. **SETUP**.
- Commands are entered in command window in bold arial font, e.g. **rts('cdcl3')**, and are executed by hitting the 'return' key

Important basic functions

- **FILE** button → allows you to view previously saved files in your default directory
- **LOAD** button → loads the FID and all acquisition parameters from the selected file
- **ABORT ACQ** → aborts/halts acquisition
- **jexpn** command → switches to experiment n (1, 2, 3, 4, 5, 6, etc.)
- **ds** → switches to interactive display mode

See Appendices 5-7 for lists of common commands and menu button options



Figure 2. In the left-hand photo an NMR user is shown adding a sample to the carousel position 1. The standby position is **ALWAYS** position 9. In the right-hand picture the computer is used to acquire, process, and display NMR data.

Logging on to Sun computer and starting VNMR

1. In order to log in enter your login ID followed by password in the welcome screen.
2. Click the VNMR icon to start the VNMR software. This looks something like **Figure 3** (see also **Figure 6**, p. 11)

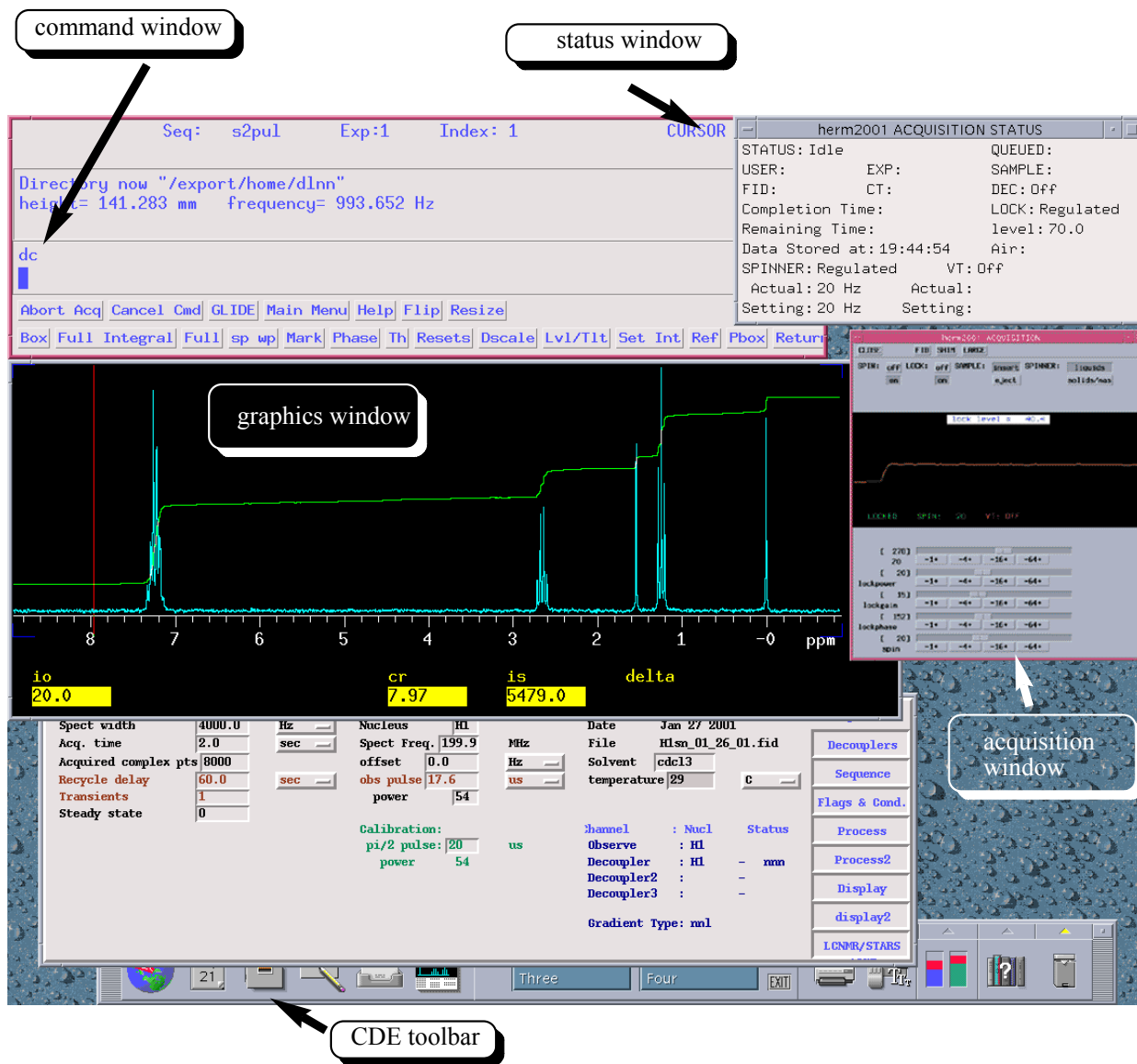


Figure 3. Layout of VNMR windows after login

Command Window: This is used to enter commands for VNMR. Also, this window contains many buttons which can be used in place of typed commands.

Graphics Window: The data, both raw and processed, and pulse sequences are displayed here.

Text Window: Parameters that have been loaded and VNMR messages are shown here. The dg interface using the label tabs (lower menu tabs) permits manual entry.

Status Window: Displays the status of any acquisition that is in progress. It includes number of scans, time remaining, sample temperature, and number of experiments queued for acquisition.

Acquisition Window: This is a pop-up window that is used to modify shims and deuterium lock parameters. This is not available during spectrum acquisition.

CDE Toolbar: This window contains the VNMR icon and the exit button. This window should appear upon login or else you have not logged in properly.

Console Window: This is normally a small icon on the desktop. It is not used except for debugging. If an error occurs be sure to write down the error message and contact NMR personnel.

Other Windows: There are other windows that are accessible. If you believe you need to access these contact NMR personnel before doing so. **ALSO NEVER EXIT VNMR FROM ANYTHING OTHER THAN THE COMMAND LINE. NOT TO DO SO CAN HARM CERTAIN FILES.**

Sample Preparation

The magnet has been shimmed on a sample with 700 μL in a 5 mm NMR tube. This corresponds to 5.25 cm of liquid volume. If you use a sample volume less than 500 μL it will be difficult to get good shimming. You will need to adjust the center line of the magnetic field with the center of the liquid level in the latter case, however.

The position of the tube should be such that the bottom of the tube is 68-69 mm from the bottom with a maximum of 70 mm for our probe design. The idea is to obtain the sample volume well centered in the center line of the coil. **Figure 2** shows an example for different tube sizes.

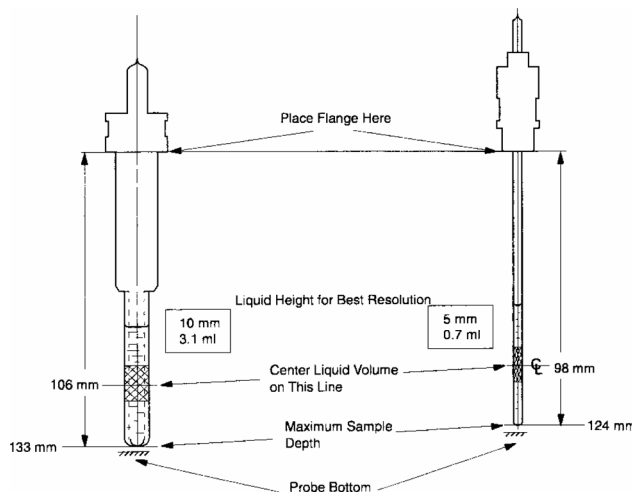


Figure 4. The correct sample position using the Varian sample depth gauge.

If you do not have 500 μL of a 1-2 mM sample you may use a Shigemi tube that require only 320 μL . The shims required for this tube will be much different from the standard shim settings, however.

The Wilmad tube code for use with the 200 MHz NMR are the 507PP type and are marked on the outside top of the tube.

How to Run 1D Experiments

Manual sample change with carousel autosampler

1. One a time sample loading requires that the ‘walkup’ mode is being used. To eject one types: **e** (ejects an existing sample). Clicking the **EJECT** button on the acquisition window to remove the sample. The carousel arm will need to be pushed to the left to release the sample.
2. Remove the sample from the spinner and replace it with your sample (use hexane and Kimwipes).
3. To insert place the sample in the magnet *make sure that the air pressure will support the sample*. If not, contact NMR personnel. Type: **i** (sample will be inserted in magnet). Clicking the **INSERT** button on the acquisition window to insert the sample.
4. If you do not hear the click of the sample resting in the probe or the indicator light does not stay on (it will blink until it reaches 20 Hz) you may have a problem with your sample. Check that you are using the correct gauging procedure.

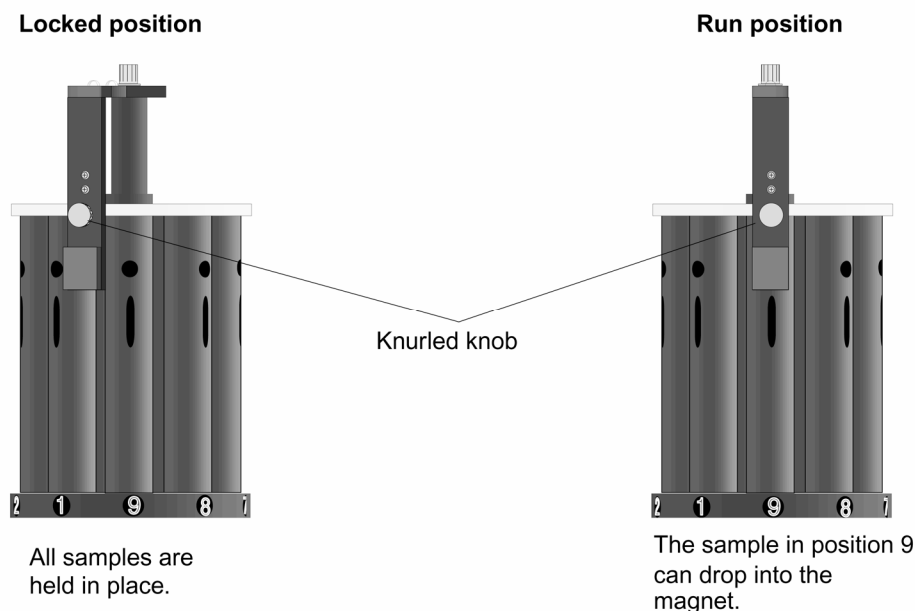


Figure 5. Carousel and sensor arm in “locked” and “run” positions, respectively.

Automated sample change with carousel autosampler

1. If the carousel autosampler is in place one must ensure that the communication link between autosampler and computer are established. The standby position should be location 9. Contact the NMR personnel if this is not the case.
2. Samples should be positioned from 1-8, carefully noting the details for your records.
3. If either the acquisition status window does not have the sample as number 9 or entering **loc?** does not return **loc=9**, in the text region above that command line, then one must enter **sethw('loc',9) loc=9**.
4. To change the sample one types: **loc=n change** (where n is the sample location). If you put your sample in position 1 then use **loc=1 change**.

Locking Samples (see Table 1) & Shimming (Field Homogeneity)

1. **jexp1** (go to experiment 1)
2. **rts('shim file corresponding to the solvent in magnet') su** will load the standard shim values that have been saved. "SETUP COMPLETE" indicates that these values have been successfully loaded. These are useful starting points to meet your sample requirements. As an example **rts('cdcl3') su** loads deuteriochloroform shims.
3. Enter **load='y' rtv('filename') su** will load the shims and parameters saved under the **svf** command.
4. Locking maintains the frequency stability. Locking makes the frequencies of the deuterium in the solvent resonances coincide with the lock frequency. The exact deuterium frequency differs from solvent to solvent and these are stored in configuration files in the computer to accommodate for these differences. Open the acquisition window by clicking the **ACQI** button. Next click the **LOCK** button and click the lock to **OFF**.
5. Change the lock power using the slide control or the -1+, -4+, -16+, and -64+ buttons. Turn up lock power and lock gain by right-clicking and turn down by left-clicking. Look for some sinusoidal variation in signal. [Note: The actual need for lock power depends on the solvent primarily. Highly concentrated deuterium signals in benzene and acetone require little lock power whereas chloroform requires much more].
6. If there is no sine wave (or just noise), click -16+ button for Z0 until such a wave appears.
7. If you know that the lock solvent has a high deuterium concentration, turn down the lock power. If the lock power is too high, the deuterium nuclei become "saturated", the signal oscillates (goes up and down), and is difficult to establish lock. The correct lock power is difficult to determine but the lock power and lock gain should be within ± 5 units of each other.
8. Adjust Z0 until a flat line appears on the sine pattern. The wavelength will increase continuously until this "square wave" pattern appears.
9. Click the lock button **ON**.
10. Adjust the lock phase to maximize the lock level.
11. Z1-Z2 Shimming
 - a. Adjust each of the following to maximize the lock signal- Z1, Z2C, Z1, Z2
 - b. Increase Z1, maximize on Z2
 - c. If the level increases, repeat step b, if lower go to d
 - d. Decrease Z1 by 20 units, maximize lock signal with Z2

- e. If level is higher, repeat step d. If the same you are finished with Z1-Z2.
12. Z3-Z4 Shimming
- a. Adjust Z3 to maximize lock. If higher go to b, if the same you are done.
 - b. Repeat Z1-Z2 shimming.
 - c. Z4 usually interacts with Z2 and so refrain from adjusting until you can predict its effect.
13. Non-spinning shims- Sometimes it may be necessary to adjust these as not all samples are the same. To determine if these need to be touched, go to the acquisition window and turn of the spinner. If the lock drops by more than 10 units, your sample might benefit from non-spinning shim adjustment.
- a. Maximize the lock by changing X. If the signal is higher continue turning the same direction until a maximum lock level is achieved.
 - b. Repeat step a with XZ shim.
 - c. Maximize the lock on the Y shim.
 - d. Repeat step c with YZ shim.
 - e. Restart spinning and readjust the spinning shims (Z1C, Z2C, Z1, Z2).

Acquisition of ¹H NMR Spectra

1. ¹H & ¹³C
- a. The Varian setup or **setup('nucleus, 'solvent')** allows you to set up a standard set of acquisition parameters without downloading the standard shim set. The macro **setup('nucleus, 'solvent')** allows you to set up standard set of acquisition parameters with solvent and observe nucleus.
e.g. **setup('H1, 'acetone')** **setup('H1, 'dms')** **setup('C13, 'C6D6')**
 - b. The message "SETUP COMPLETE" means that all parameters have been loaded.
 - c. Type **dg** to display parameters ("display guide")
 - d. To change a parameter, type the abbreviation for the parameter, an equal sign, and the new value. For example, to change the number of transients (pulses) (**nt**), delay time (**d1**), and pulse width (**pw**), type
nt=4 d1=5 pw=7 <return>
 - f. Type **ga**. Wait and watch for ct (completed transients) of 2 or more, type peek, to examine the spectra obtained. If you want to change the number of transients at this point type **sa nt=32 ra** (**sa** is stop acquisition, **ra** is resume acquisition).
2. Acquiring ³¹P & ¹⁹F Data
- The procedure here is analogous to that above but there must be some routine probe tuning and other hardware changes. Please consult the NMR personnel if you need additional training to obtain these data.
3. Data Acquisition using tab buttons
- Alternatively you can use the tab buttons in the windows that are displayed by clicking on **SETUP** Menu buttons. These are located both in the upper command window area and also in the lower text window areas (see **Figures 5 and 6**).

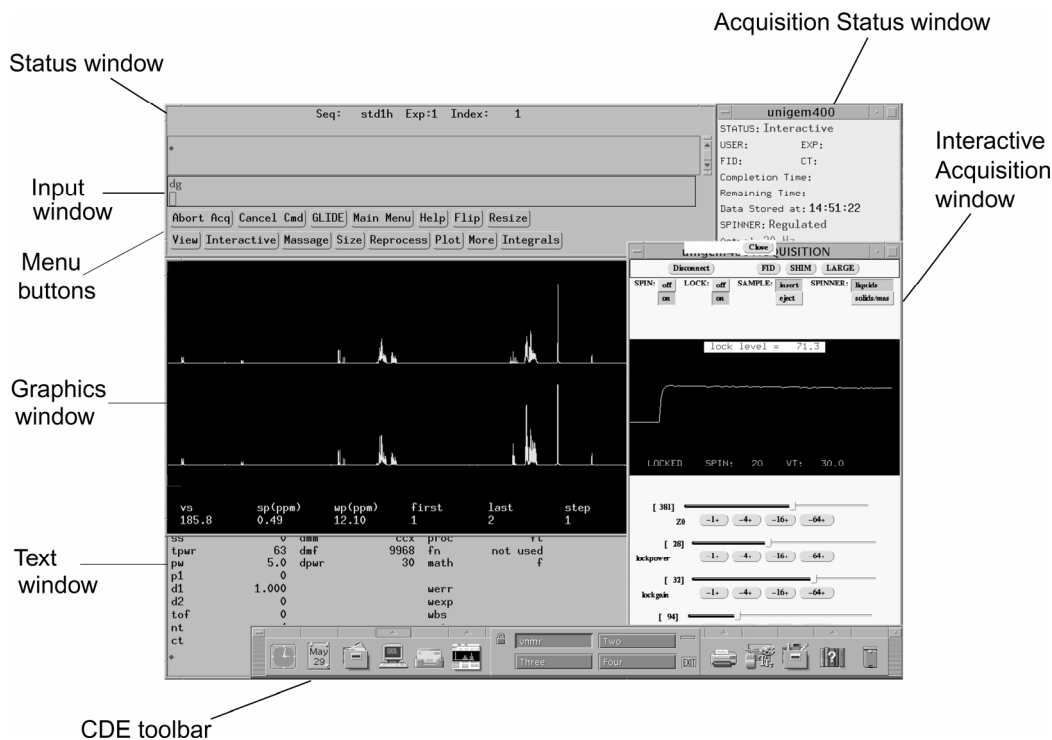


Figure 6. Upper command area showing menu buttons and assorted windows.

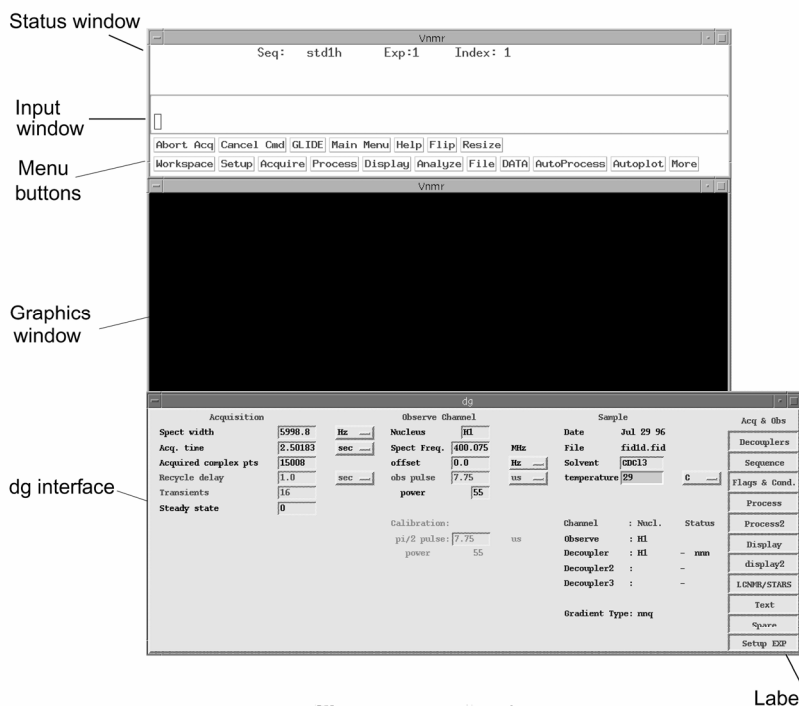


Figure 7. Upper command and lower text window area showing SETUP buttons.

The **SETUP** buttons are located in both upper and lower windows in the Menu and Label tabs, respectively. From these one can navigate through experiment setup, acquisition and processing options (see Appendix 7, Menu Tab Tree).

4. Decoupling and Non-decoupling Experiments

Proton decoupling is done frequently to simplify and enhance X-nucleus spectra. It is possible to use Waltz-16 broadband decoupling to eliminate the effects of proton coupling to carbon, for example. To do this no pulse programming is required but simply telling the experiment when to turn on the decoupling through the decoupler mode command (**dm**). There are several situations:

dm = 'yyy' (decoupler always on; decoupled with NOE)

dm = 'nny' (decoupler gated off prior to pulse/acquisition; decoupled without NOE if delay is 5x longest T_1 ; useful for quantitative ^{13}C)

dm = 'yyn' (decoupler gated off during acquisition; not decoupled but with NOE)

dm = 'nnn' (decoupler always off; not decoupled and without NOE)

One can also perform selective and off-resonance decoupling by changing the decoupler modulator mode (**dmm**). Here **dmm='c'** is used as opposed to **dmm='w'** to mean continuous wave decoupling vs. waltz. If such experiments are planned there may need to consultation with NMR personnel for help with determining decoupler power.

Processing Data on the Sun

The **ft** command Fourier transforms one or more FIDs without weighting being applied to the FID. To perform the FT with weighting use the **wft** command. Weighting or apodization functions will be discussed in more detail as you study signal processing in chemistry. Here you need to understand that an exponential multiplication is being performed in the **wft**. The result is signal-to-noise enhancement with generally some loss in resolution possible. You can experiment with various weights (**lb** command and **ADJ WEIGHTING**) to examine these effects.

For the FT process the **fn** parameter is used that must be a multiple of two; for example, 16384, 32768, or 65536 (or written as 16k, 32k, or 64k, k being a multiple of 1024). Typically **fn='n'** such that **n** becomes however many data points were collected (**np**) and the first power of 2 will be the value of **fn** used, the remaining being filled with zeros. Thus, zero-filling for digital resolution enhancement is done by choosing a larger **fn**, typically twice as large, than **np**.

1. Phasing Spectra

Doing a complex FT entails producing two sets of data, referred to as the cosine and sin parts, or real and imaginary data sets. Usually the spectral data must be created as linear combination of these data sets. The problem is find the angle that can be used to mix the two data sets to produce the experimental data where all peaks are properly in-phase, i.e. standing up straight.

This is complicated by the fact that this angle Θ is a function of frequency:

$$\Theta = \mathbf{rp} + (\omega - \omega_0) \times \mathbf{lp}$$

Here **lp** is left or first-order phase and **rp** is right or zero-order phase constants. The **rp** parameter is frequency-independent and **lp** is frequency-dependent. To adjust **lp** and **rp** the following is done:

- They can be entered like any parameter, e.g. **lp**=-180
- The process can be done automatically using the **aph** command.
- Manual adjustment is possible if the results from **aph** are not acceptable (demonstrate)
 - Click the **PHASE** button
 - Click on the screen on the right side of the spectrum
 - Move the cursor up and down to adjust **rp** and adjust the phase of the peak near the cursor
 - Click on the left side and adjust **lp** in the same manner
 - When finished type **ds**

2. Referencing Spectra

- a. To view the axis type **dscale**. To alternate displays between Hz and ppm use **axis='h'** or **axis='p'**.
- b. Click the button Cursor button, once for one cursor, twice for two cursors, and a third to remove cursors.
- c. Get a cursor to move to TMS.
- d. At the command line type: **nl rl(0p)** or click the button **REF** to reference TMS or other reference with the mouse.

3. Integration

- a. Display the full spectrum using **ds full**.
- b. Clicking the **INTEGRATE** button permits integration through mouse and menu buttons.
- c. The **SET INT** button permits referencing of integrals to a set value.

4. Peak Picking

- a. Type **ds**.
- b. Click **TH** button to activate the peak threshold.
- c. Adjust the height of the threshold line using the mouse to select all peaks above this line. Type **dppf** to display the peak frequency.

5. Insets

The **inset** command displays the part of the spectrum between the two cursors using **ds** from the command line. The vertical position of the inset is shifted up about one-quarter of the height of the whole spectral display window. The original “full” spectrum remains on the screen but the parameters on the bottom of the display pertain to the inset spectrum. If desired the integral display and axis scale are also shown in the inset.

After running inset, you can modify the spectrum with the mouse buttons (expand, for example) or using the menu options. The mouse buttons function as follows:

- The left mouse button positions the cursor or pair of cursors. In the sc wc mode the left buttons controls the starting position of the display. In the sp wp mode, the left button controls the starting frequency.
- The center mouse button changes the vertical scale of either the spectrum or integral. Clicking or rolling the mouse while depressed permits an increase in vertical scale (**vs**).
- The right mouse button positions the second cursor relative to the first cursor. In the sc wc mode the cursor displays the width of the display. In the sp wp mode it controls the range of frequencies displayed.

These features may be demonstrated for plotting spectrum under VNMR but routinely see below for off-line printing from a PC in SB 454:

Text or titles:

To create use the command line: **text('comments\\comments\\comments')**
produces:

```
comments
comments
comments
```

Plotting text only: **pltext**

Plotting spectrum use the command line: **pl**

Plotting scale: **pscale** (recall **axis= 'h'** for frequency scale)

Plotting an integral: **pir**

Plotting most all parameters: **pap** (use **ppa** to print basic parameters)

Plotting peak frequencies above threshold (**th**): **ppf** (to display of frequencies use **dpf**)

Plotting peak listing: **pil**

These features will be demonstrated for plotting spectrum on a PC in SB 454 using the ACD processing software:

The above steps 1-5 can all be accomplished using the ACD 1D NMR processing software (follow flash demo movie links below for additional training).

Processing: http://www.acdlabs.com/download/movies/flash/1dnmr_1_flash.html

Reporting: http://www.acdlabs.com/download/movies/flash/1dnmr_2_flash.html

Logging off the spectrometer

1. Re-lock the spectrometer after you are finished by inserting the standard lock sample (0.1% ETB in CDCl_3). The carousel should be in position 9 when finished.
 - a. Type **e** from the command line or use EJECT from the menu button in the acquisition window to eject sample.
 - b. Move the carousel to position 9 if needed. Type: **loc='9' change**.
 - c. Type **i** from the command line or use INSERT from the menu button in the acquisition window to insert the standard lock sample.
 - d. Lock the sample.
 - e. Type **exit** at the command line.
 - f. Click exit on the CDE toolbar.
 - g. Sign out in the log book.

Notes

Appendix

1. **Checking acquisition progress:** To stop the acquisition progress type **sa**, use **nt** to increase the number of transients, and then **ra** to resume starting with last one stopped after the **sa** command.
2. **Saving data to disk:** Use **svf('filename')** [Use a valid UNIX filename with alphanumeric characters only (letters, numbers, periods, hyphens and underscores) and NO spaces]
3. **Table of lock frequencies for the Mercury 200**

Solvent	Z0	Lock power	Lock gain	Lock Phase	Date
Acetone-d6					
CDCl3					
CD2Cl2					
CD2ClCD2Cl					
CD3OD					
CD3CN					
C6D6					
CD3NO2					
DMSO-d6					
THF-d8					
Toluene-d7					
D2O					

4. Table of Common Troubleshooting Solutions

Commonly Encountered Problems by users of the Mercury-200	SOLUTIONS
Paper tray Empty	Go to the PAPER cabinet and fill the paper tray in the plotter.
Sample not spinning	1) If you are on an R2D2 magnet, you may have to "help" set the sample in the probe. To do this, cover the transfer tube opening in the center of the magnet briefly with one hand and wait for the spinner light to turn on. 2) If the sample still refuses to spin check on the computer terminal in lock or shim display and verify the spinning is turned ON. 3) Check the command spin='y' not spin=0 or 'n' in the vnmr command line.
Broken tube inside the magnet	STOP!!! Sample inserted with no spinner! Sample with spinner dropped too fast Call the NMR staff immediately.
NMR tube slips too easily on the spinner	O-ring needs to be replaced. Call the NMR staff ASA
Frozen Screen	COMPUTER HAS CRASHED. Call the NMR staff ASAP. DO NOT TURN THE UNIX STATION OFF!
UNABLE TO LOCK SAMPLE	1) LOCK POWER LEVEL TOO HIGH. 2) Z0 OFF RESONANCE. 3) FORGOT TO ADD 2H SOLVENT. 4) UPLOAD STANDARD SET OF SHIMS. 5) CHECK THE VALUES OF Z ₀ IN TABLE OF LOCK SOLVENTS.

5. Quick VNMR Sheets

Login

- (1) Sign into Spectrometer and Business Office Log Books.
- (2) Sign in to the spectrometer using your assigned Username and Password.

Sample

- (1) [eject] [insert]

Tuning The probe does not need to be tuned. DO NOT ATTEMPT TO TUNE THE PROBE IN THIS INSTRUMENT!

Load Shims

rts('shim file corresponding to probe in magnet') su

Locking Adjust lock power, lock phase, lock gain, and z0 (check for saturation of lock)

Shimming SHIM CONTROLS-- z1c z2c z1 z2 z3

Acquisition Select SET UP tab or use **setup('nucleus', 'solvent')** to set up parameters for acquisition.

Where *nucleus* is a 'H1', 'F19', 'C13' or 'P31'; and *solvent*.

FLAGS-- in il dp wexp wbs wnt werr

Processing

lb = 1.0

wft

Saving and Retrieving Data

VNMR COMMANDS:

svf('filename') rt('filename') svp('filename')

explib jexpn cexp(n) delexpn

Note: n = 1-9

6. VNMR Commonly Used Keyboard Commands

<u>TAB Button</u>	<u>KEYBOARD COMMAND</u>	<u>DESCRIPTION</u>
<u>ACQI</u>	acqi	Opens acquisition window
<u>Eject</u>	e	Ejects the lock sample tube and spinner turbine from the magnet.
<u>INSERT</u>	i	Inserts your sample and spinner turbine into the magnet.
	rts ('cdcl3') su or rts ('c6d6') su	Retrieve shims from the shims directory. Set up or load the shim values into the program. To see a list of shims, enter dgs which means display graphic shims.
<u>LOCK</u>		Activate lock screen. Set the spin to 20 revolutions/sec. Set the phase angle, set the power in range of 25-35 decibels for proton, set the gain (set at maximum if desired), search for resonance with Z0 (offset).
<u>SHIM</u>		Activate shim screen. Adjust Z1C, Z2C, Z1, Z2, Z3 shims to improve magnetic field homogeneity.
<u>SETUP</u>		Displays the nucleus and solvent menu.
<u>H1, CDCL3</u>	setup('nucleus', 'solvent') where the nucleus is 'H1', 'F19', 'C13', or 'P31'.	Parameter setup. Selects the default parameters for the desired nucleus and solvent. Wait approximately 20 seconds. See message example: "H1 setup complete" and see list of default acquisition parameters. To see a list of acquisition parameters in other situations, enter dg which means display guide.
<u>C13, CDCL3</u>		
<u>F19, CDCL3</u>		
<u>P31, CDCL3</u>		
<u>NUCLEUS, SOLVENT</u>		
<u>SELECT ONLY ONE</u>		
	nt? nt=4	What is the number of transients? Computer will answer. Set the number of transients to 4.
<u>ACQUIRE</u>		Displays the menu to start the acquisition of data.
<u>GO, WFT</u>	go wft	Acquire data with automatic weighted Fourier transform. 1. Transmit pulse and receive FID (free induction decay) 2. Weighted Fourier transform 3. Display spectrum. (The height can be adjusted by placing the arrow cursor on the desired peak and then holding down the center mouse button and dragging.) To stop acquisition type sa

wysiwyg = 'n'	Selects the adjacent plotter. If this command is not entered, the printed spectrum maybe less than a full page. To get the spectrum to fill the page, it maybe required to enter wcmx=250 wc2mx=180
f full	Display full spectrum on a full screen. This is only necessary if you think some portion of your spectrum is not being displayed. This can be used on inset command if the user wishes to return to a full spectrum display.
<u>PHASE</u> If this tab does not appear try typing ds at the command line. See under <u>DISPLAY INTERACTIVE</u>	Manually phase spectrum.
aph	Automatic (spectrum) phase.
<u>DSCALE</u> To find tab go to <u>DISPLAY</u> and <u>INTERACTIVE</u>	
dscale	Display scale
axis = ('p') or axis = ('h')	Change the scale values to either ppm or hertz
nl	Sets the cursor precisely to the nearest peak. Repeat if necessary, see message "Height = XXmm Frequency=XXppm".
rl (x.xxp)	Set the frequency of the peak at the cursor as the reference line. The frequency = X.XXppm, for TMS set to 0.00p.
<u>PART INTEGRATION</u>	See green solid integral plot on the screen.
<u>RESET</u>	Move the arrow pointer to the left side of the left peak then left click. Move the arrow pointer to the right side of the peak and left click. Repeat for all peaks except for TMS. This resets the dc level to zero when the command bc (baseline correct) is typed..
cz	Clear integral of resets, only necessary if errors were made above.
dli	Display the list of integrals. Select <u>FLIP</u> to see the list below the spectrum.

SET INT

Integral normalization scale. When viewing the list of integrals, select the one which represents a specific number of protons=N (example CH₂, N=2). Place the vertical red line cursor at the center of the desired peak (enter nl) then select SET INT. See the message "current integral is 0.00. New value?" Enter the number of protons (example 2). Select FLIP to see new list.

dpir Displays on spectrum window the number and location of protons. $V_p \geq 12$ for this command to operate.

ds Display the spectrum

EXP--

To find, go to WORKSPACE
current EXP is not shown.

jexp# Join experiment #X, to see all experiments in memory type **explib**

dpf Display the peak frequencies. Adjust the number of peaks identified by selecting th. See the yellow horizontal line across the screen. Place the arrow cursor on the threshold line and drag up (to decrease) or down (to increase) the number of peaks located. Repeat dpf as desired.

ppf Print the peak frequencies.

pl pscale Plot spectrum, plot scale. This represents two commands separated by a space.

text ('training gemini 200') This puts text into computer memory and displays it on the screen.

pltext or pltext (200, 600) Print text. Usually in the upper left corner of the paper.
pltext (vertical position, horizontal position)

inset Separates a small portion of the spectrum for expansion and repositioning. Initially user must select the area to be copied by placing a vertical red cursor line on each side of the selected area. If necessary type ds to get the cursors to appear or go to DISPLAY and INTERACTIVE.

pap Print all parameters.

ppa Print the (most critical) parameters.

ap Print all acquisition parameters

page

Instructs the printer to immediately begin printing all print jobs that are waiting in the queue.

(Example: pl pscale ppf pir page)

exit

When your experiment is complete

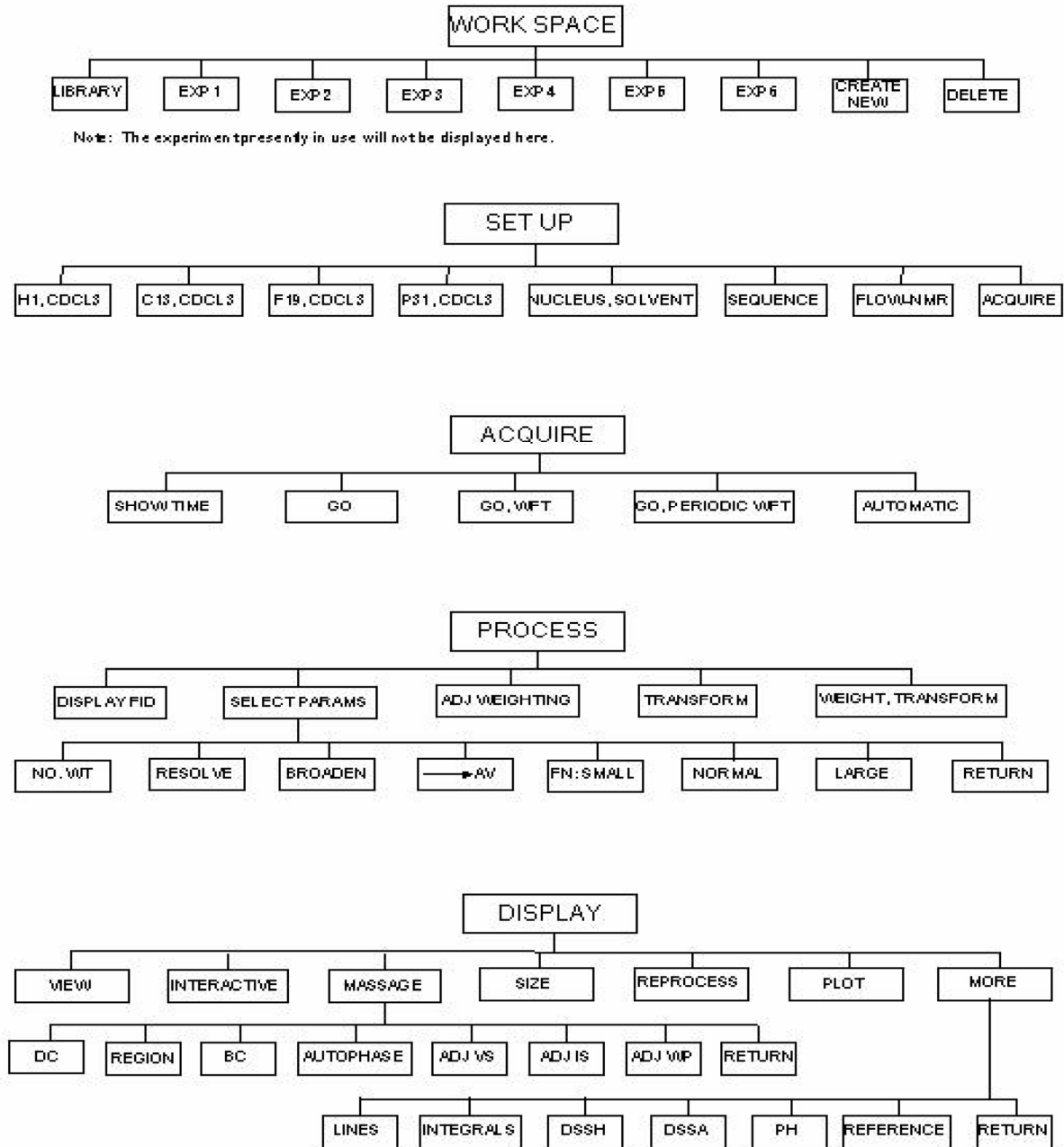
1. remove your sample
2. put the "lock" sample in the magnet and make sure the spectrometer is "locked"
3. type exit on the command line (very important)
4. select exit on the Unix desktop screen

exit is a macro command that

1. resets the room temperature shims for the lock sample
2. turns off all the decoupling pulses and makes the spectrometer safe so it will not destroy itself

7. Menu Tab Tree

MERCURY TABS



8. Checkout Practical Exam Requirements

Prior to performing this practical exam you must first pass a written exam. The written exam tests you on material in this document, as well as spectrometer facility matters.

This is an outline of the tasks you will be required to perform correctly in order to pass the checkout practical exam. You will be allowed up to 30 minutes 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. IPFW staff may, at their discretion, observe you performing all or part of the checkout exam.

Part A. Test Sample – 10% Ethylbenzene in CDCl₃ (or other sample as required)

- login to computer and start VNMR
- remove CDCl₃ standard, insert new sample, lock and shim
- run ¹H NMR spectrum, process, integrate and plot
- set up new experiment for ¹³C NMR
- run ¹³C spectrum, process and plot

Part B. Standard Sample – 0.1% Ethylbenzene in CDCl₃

- remove test sample and replace with 0.1% ETB standard, lock and shim
- initialize spectrometer to ¹H
- shutdown VNMR and logout from UNIX

Name of colleague _____

Date of practice _____

More Advanced Training

Attached proton test (APT)
90 degree pulse width calibration
DEPT
COSY, NOESY, TOCSY
HMBC, HMQC
Inversion decay for T₁ measurement, Kinetics