Varian NMRs
Mercury 400, 400MR, 400SGM, VNMRS-500, VNMRS-600

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We currently have five Varian NMRs. The Mercury 400 runs with VnmrJ 2.2D and VnmrJ 3.2A is on the others. The NMRs have a full range of experiments that are described later. Each NMR has some different capabilities which are described below.

Varian Mercury 400:
The Varian Mercury 400 is a fixed nucleus system. It has a two channel indirect probe tuned to ^{1}H and ^{13}C. The indirect design gives an excellent ^{1}H sensitivity and also excels at the 2D $^{1}H\{^{13}C\}$ experiments like HSQC and HMBC.

Varian 400MR:
The Varian 400MR NMR is a fully automated system. It has a two channel broadband probe which will autotune itself. The high-band channel does ^{1}H and ^{19}F. The low-band channel will tune to any nuclei that falls between ^{31}P and ^{15}N. If you need to run a nuclei other than ^{1}H, ^{19}F, ^{13}C, or ^{31}P, see Allan Kershaw to have the needed nuclei added to your experiment list.

Varian 400SGM:
The Varian 400SGM NMR is a fully automated system identical to the 400MR. It is primarily used for undergraduate classes, but can be used by others if needed when it is not being used for class work. It has a two channel broadband probe which will autotune itself. The high-band channel does ^{1}H and ^{19}F. The low-band channel will tune to any nuclei that falls between ^{31}P and ^{15}N. If you need to run a nuclei other than ^{1}H, ^{19}F, ^{13}C, or ^{31}P, see Allan Kershaw to have the needed nuclei added to the experiment list.

Varian VNMRS-500:
The Varian VNMRS-500 NMR is a fully automated system. It has a two channel broadband probe which will autotune itself. The high-band channel does ^{1}H and ^{19}F. The low-band channel will tune to any nuclei that falls between ^{31}P and ^{15}N. If you need to run a nuclei other than ^{1}H, ^{19}F, ^{13}C, or ^{31}P, see Allan Kershaw to have the needed nuclei added to your experiment list. The VT range is -80°C to 100°C.

Varian VNMRS-600:
The Varian VNMRS-600 NMR is our top end instrument. There are two probes available. The default probe is a two channel broadband with full autotune. The high-band channel does ^{1}H and ^{19}F. The low-band channel will tune to any nuclei that falls between ^{31}P and ^{15}N. If you need to run a nuclei other than ^{1}H, ^{19}F, ^{13}C, or ^{31}P, see Allan Kershaw to have the needed nuclei added to your experiment list. The VT range for this probe is -80°C to 100°C. The other probe is a three channel probe set for ^{1}H, ^{13}C and ^{15}N. As such, it is ideal for protein sample analysis. The VT range is for this probe is -20°C to 80°C.

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Windows:
The VnmrJ screen has many areas to work with. See the graphic on the next page for details.

Menu Bar	Windows type Menu system for access to various functions
System Toolbar	A variety of commonly used system functions
Experiment Selection	List of available acquisition experiments
Study Queue	Display area showing what experiments are queued up
Display Screen	Shows spectral data
Graphics Toolbar	Functions to control the Display Screen
Parameter Area	All parameters may be accessed via menu on left side

Conventions:

Italics	You type this on keyboard (VNMR Command Line Window needs to be active if using VNMR command)
Boldface	The specified VnmrJ button is clicked (Generally use left button)
<Tab>	Tab pointer

Important General Rules and Criteria:
All NMR tubes used in these instruments must be Wilmad 535 or Wilmad 1235 tubes. Anyone found using tubes of lesser quality may be removed from the access list of these instruments. Do not oven dry these NMR tubes. Doing so will change the specifications on these tubes to lower values and instrument damage may result.

Sample volume should be 5cm in height. Lower volume may lead to weak lock and/or auto-shim failure. Manual shimming will be required.

Do not modify, add or remove material form instrument standards. They are set up for specific operations. Contact Allan Kershaw if you feel that the standard is not usable.

If there is a problem with the instrument, do not try to fix it yourself. Note the error in the logbook and email me. Uninformed attempts to fix the problem usually lead to bigger and more serious problems.

Do not intentionally damage or deface any part of the instrumentation facility. This includes the instruments, related computer monitors, keyboards and mice and the instrument logbook.

What to do if you break a sample or a plug tube:

1. Do not panic or try to hide the problem. Accidents happen and it is important to safely clean up any spill so other users are not harmed and the instrument is not damaged further.
2. Record the incident in the logbook.
3. Do not try to clean the spinner or any NMR components contaminated by the spill. Do not remove any broken tube remnants from the spinner. Place the spinner and all glass fragments on the sink counter.
4. Try to contact the system administrators, Allan Kershaw or Dr. Travis Williams. Phone numbers are posted in the NMR room.
5. If you can not contact either, send them both an e-mail. Mark the instrument as unusable so other users will not compound any damage. You are responsible for the instrument until a system administrator shows up and takes control.
6. If you find an instrument marked with a problem, do not use it. If you fail to heed such a warning you may be financially responsible for any damage to the instrument.

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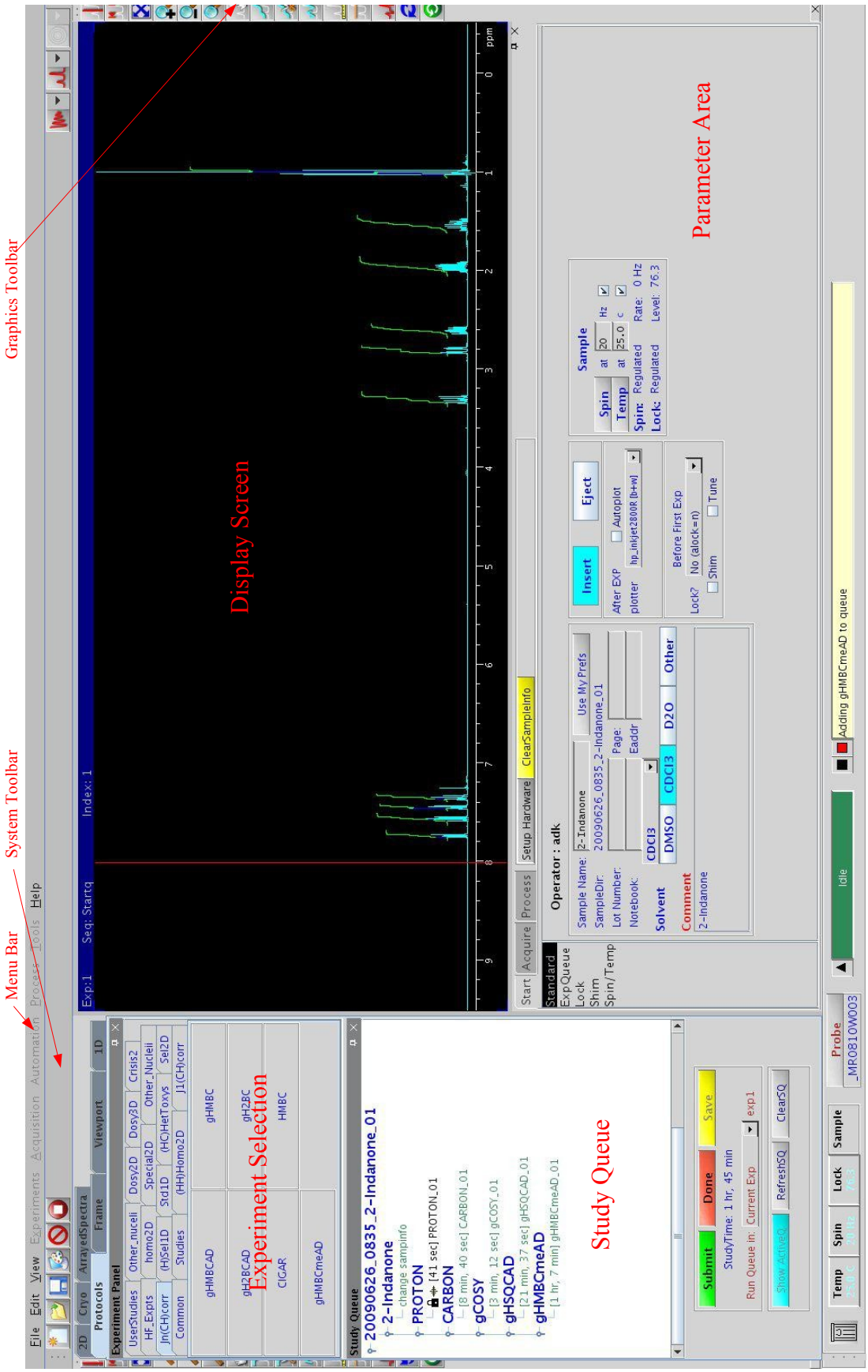

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For 400SGM Sample Changer Operation – skip two pages

Log in on Instrument:
Sign in on the logbook with the date, your name, a contact phone number and the time you start.
username
password
Double-click the VnmrJ ICON on desktop (looks like NMR spectrum)

Running a Sample:

1 **New Study**	Starts queue set up. Display sample information. Do not 'Double Click' this button.
2 **<Start>**	
2.1 **Standard**	
2.1.1 *Name Sample*	Enter sample name (Required)
2.1.2 **Select Solvent**	Use drop-down box to select other solvents
2.1.3 **Eject** and **Insert** Sample	Carefully remove the sample from the spinner. Insert new sample into spinner until it touches the bottom of the depth gauge. Do not touch the sides of the spinner. Do this away from the table so if a spill occurs, it will not contaminate the table surface.
2.1.4 **Set Before First Exp**	Options for Auto Lock, Tune, and Gradient Shim
3 **<Protocols>**	Experiment Selection area
3.1 Select all desired experiments	Click experiment buttons to add all desired experiments (See Experiment List for descriptions) Recommend that you start with Proton only first and then add additional experiments as described below.
4 **<Acquire>**	When finished, click time point to modify any parameters
4.1 **Default**	
4.1.1 Set spectral width	
4.1.2 Set Number of Scans	
4.1.3 Set Relaxation Delay	
4.1.4 Set H1 Dec. Mode	Only for experiments where an X nuclei is being observed.
4.2 Repeat for each experiment selected	
5 **Submit**	Start study queue. System will do all selected experiments.
6 **<Process>**	
6.1 **Autoprocess**	When the block size is reached or the experiment is completed, this will show you the results.
7 **Continue Study**	If abouve results are good, then reload current study queue
1.1 Select additional experiments	Select any additional experiments and modify parameters as described above
1.2 **Submit**	Restart current study queue at next experiment

Addition Useful Commands:

1. **Stop**	Stops acquisition of current experiment and all subsequent experiments. Current data set will have data as of last BS.
2. **File – Auto Save**	Save your data as it currently is using the Chempack directory structure

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Log off Instrument:

1 Reinsert standard sample
2 **<Start>**
3 **Standard**
  3.1 **CDCl3** Set solvent to Chloroform
4 **Lock**
  4.1 **Gradient Shim**
  4.2 **Find z0**
  4.3 **Spin on**
5 Standard must have a stable lock level of 75 or more before logging off.
6 **File – Exit VnmrJ**
7 **Log Out Icon**
8 Sign out on the logbook with the time you have finished and record which experiments you ran

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Running samples on the 400SGM with the 7600 Autosampler

400SGM Sample Changer Specific Criteria:

Failure to follow these criteria can lead to failed sample run and possible damage to your NMR tube and the Sample Changer

- Sample volume must be 5cm or higher.
- NMR tube must touch bottom of depth gauge.
- Top of NMR tube must not extend more than 8cm above spinner top. Check carefully with J-Young tubes.
- No valves, label tags, etc. may project more than 1cm from NMR tube wall.
- Deuterated lock solvent must be used. Solvents with multiple Deuteron signals may fail.

Important things to know:

- When a command is active, the blue experiment bar will show with diagonal bars.
- Do not attempt to access or modify any experiment and sample once it has been submitted.
- Experiment time is limited to 60 minutes total in the Day Queue. Any experiments exceeding this time will be shifted to Night Queue, which will run after 20:00. Night Queue limit is 12 hours
- Do not process data on these consoles. This can lead to failure of queue.
- Do not shut off VNMRJ. There is no 'Exit' in menu. Do not use the 'X' button in upper right corner.

Log in on Instrument:

In the logbook, record the date, your username, a contact phone number, the time you submit the sample, and the location(s) used.
Select username from list
password
OK

Placing and Removing Sample in tray:

1. Press white 'Access Request' button located on Sample Changer panel
2. Wait for lock to open
   Do not open the door until the door is unlocked.
   This may take several minutes if the changer is in operation
3. Open door
4. Select spinner, remove plug and place in tray
5. Place sample in spinner
6. Make sure sample tube is touching the bottom of the spinners and does not exceed maximum sample height
7. Return spinner to its original location
8. Note location for recording in logbook
9. Close door

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Running a Sample:

1	**New Study**	Starts queue set up. Display sample information. Do not 'Double Click' this button.
2	Enter Sample Information	
	2.1 *Name Sample*	Enter sample name (Required).
	2.2 **Select Solvent**	Use drop-down box to select other solvents.
3	Select Experiments	Experiment Selection area.
	3.1 Select all desired experiments	Click experiment buttons to add all desired experiments (See Experiment List for descriptions).
4	**Experiment Name**	When finished, click time point to modify any parameters.
	4.1 Set spectral width	
	4.2 Set Number of Scans	
	4.3 Set Relaxation Delay	
	4.4 Set H1 Dec. Mode	Only for experiments where an X nuclei is being observed.
	4.5 Repeat for each experiment selected	
5	**Submit**	Check for location.
	5.1 **Select location** locations	Click location where sample was placed. Multiple may be selected using CTRL - Click.
	5.2 **Submit**	Save study to queue. Study will be run in turn.
6	**Done**	Close current study (Wait until 'Locations Accepted' shows).
7	**Logout**	Click to exit.
8	Remember to retrieve your sample after the the run has been completed	
	8.1 Remove your sample tube	
	8.2 Place plug in spinner	
	8.3 Put spinner back in empty location	
	8.4 Record the time you removed your sample in the logbook	

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CHEMPACK Experiment List

If you do not know and/or understand all of the parameters used by these experiments, then please discuss this with Allan Kershaw before you attempt to run them. Improperly set parameters could lead to long acquisitions with no results. Some of the experiments listed below are not available on the Mercury 400 due to limitations in the older console (indicated by * in first column).

Common

	PROTON	Standard ^{1}H acquisition
	CARBON	Standard ^{13}C acquisition
	PRESAT	Standard ^{1}H acquisition with presaturation pulse for solvent suppression
	wet1D	Standard ^{1}H acquisition with Wet pulse for solvent suppression
	(HH) gCOSY	^{1}H - ^{1}H chemical shift correlation with gradient cycling
*	(HC) HSQCAD	^{1}H - ^{13}C chemical shift correlation, HSQC using adiabatic pulse sequence
*	(HC) gHMBCAD	^{1}H - ^{13}C chemical shift correlation, gHMBC using adiabatic pulse sequence
	(H) NOESY1D	^{1}H - ^{1}H chemical shift exchange using phase cycling, 1D version

Std1D

	PROTON	Standard ^{1}H acquisition
	CARBON	Standard ^{13}C acquisition
	(H) T1-Measure	Measure T1 of ^{1}H signals
	(H) T2-Measure	Measure T2 of ^{1}H signals
	(C) APT	^{13}C acquisition to determine C-H multiplicity using variable pulse widths
	(C) DEPT	^{13}C acquisition to determine C-H multiplicity using polarization transfer
*	FLUORINE	Standard ^{19}F acquisition
*	PHOSPHORUS	Standard ^{31}P acquisition
	Pureshift 1D	^{1}H-decoupled ^{1}H acquisition
*	C_T1_Measure	Measure T1 of ^{13}C signals

Studies - These are composite commands that call multiple experiments

H1COSY	PROTON – gCOSY
H1C13	PROTON – CARBON
H1COSYQC	PROTON – gCOSY – HSQCAD
H1COSYQCBC	PROTON – gCOSY – HSQCAD – gHMBCAD
C13APT	CARBON – APT
C13DEPT	CARBON – DEPT

(HH)Homo2D

	gCOSY	^{1}H - ^{1}H chemical shift correlation with gradient cycling
	COSY	^{1}H - ^{1}H chemical shift correlation with phase cycling
	gDQCOSY	^{1}H - ^{1}H chemical shift correlation using double quantum filter and gradient cycling
	DQCOSY	^{1}H - ^{1}H chemical shift correlation using double quantum filter and phase cycling
	TOCSY	^{1}H - ^{1}H total chemical shift correlation using coherence transfer
*	zTOCSY	
	NOESY	^{1}H - ^{1}H chemical shift through space correlation using phase cycling, signals are pos/neg
	ROESY	^{1}H - ^{1}H chemical shift through space correlation using phase cycling, all signals are positive
*	ROESYAD	^{1}H - ^{1}H chemical shift, ROESY using adiabatic pulse sequence

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J1(CH)corr

*	HSQCAD	^{1}H - ^{13}C chemical shift correlation, HSQC using adiabatic pulse sequence
*	gHSQCAD	^{1}H - ^{13}C chemical shift correlation, gradient version of HSQCAD
	HSQC	^{1}H - ^{13}C chemical shift correlation using phase cycling with multiplicity detection
	gHSQC	^{1}H - ^{13}C chemical shift correlation, gradient version of HSQC
	HMQC	^{1}H - ^{13}C chemical shift correlation using phase cycling with no multiplicity detection
	gHMQC	^{1}H - ^{13}C chemical shift correlation, gradient version of HMQC
	HETCOR	^{1}H - ^{13}C chemical shift correlation using ^{13}C detection
	gHETCOR	^{1}H - ^{13}C chemical shift correlation, gradient version of HETCOR
*	ASAPHMQC	^{1}H - ^{13}C chemical shift correlation, high speed acquisition version of gHMQC

Jn(CH)corr

*	gHMBCAD	^{1}H - ^{13}C chemical shift correlation, gHMBC using adiabatic pulse sequence
	gHMBC	^{1}H - ^{13}C long range chemical shift correlation, gradient version of HMBC
*	gH2BCAD	^{1}H - ^{13}C long range 2-bond chemical shift correlation using adiabatic pulse
	gH2BC	^{1}H - ^{13}C long range 2-bond chemical shift correlation
	CIGAR	^{1}H - ^{13}C long range chemical shift correlation with 2 and 3 bond distinction
	HMBC	^{1}H - ^{13}C long range chemical shift correlation using phase cycling
*	*gHMBCmeAD	

(H)Sel1D

	NOESY1D	1D version of NOESY
	ROESY1D	1D version of ROESY
	TOCSY1D	1D version of TOCSY
*	zTOCSY1D	1D version of zTOCSY
	selexcit	
*	stepNOESY1D	

(HC)HetToxys

*	HSQCADTOXY	^{1}H - ^{13}C total chemical shift correlation using adiabatic pulse with multiplicity detect
*	gHSQCADTOXY	^{1}H - ^{13}C total chemical shift correlation, gradient version of HSQCADTOXY
	HSQCTOXY	^{1}H - ^{13}C total chemical shift correlation using phase cycling with multiplicity detect
	gHSQCTOXY	^{1}H - ^{13}C total chemical shift correlation, gradient version of HSQCTOXY
	HMQCTOXY	^{1}H - ^{13}C total chemical shift correlation using phase cycling
	gHMQCTOXY	^{1}H - ^{13}C total chemical shift correlation, gradient version of HMQCTOXY

Sel2D

* (HC) bsHSQCAD
* (HC) bsgHSQCAD
* (HC) bsgHMBC
* (HC) EXSIDE
* (HH) bsTOCSY
* (HH) bsNOESY
* (HH) bsROESY
* bsHSQCNOESY
* bsHSQCROESY

HF_Expts – Not Available due to hardware limitations

* Hobs_Fdec
* Fobs_Hdec
* FH_HOESY
* FH_gHETCOR

Dosy2D – 2D *D*iffusion *O*rdered *S*pectroscop*y*

* Used to for studying diffusion coefficients in mixtures

Dosy3D – 3D *D*iffusion *O*rdered *S*pectroscop*y*

* Used to for studying diffusion coefficients in mixtures

Crisis2 – *C*ompensation of *R*efocusing *I*nefficiency with *S*ynchronized *I*nversion *S*weep

* Pulse sequence for improved sensitivity and selectivity in 2D $^{1}H - ^{13}C$ sequences

Useful Macro

* decnuc — Usage: decnuc('nuc') Loads decoupling parameters. Nucleus must be from opposite frequency band. See ADK if parameters do not exist.

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SIGN UP POLICY FOR THE Varian Mercury 400 and the Varian VNMRS 500 NMR Instrument
Every day of the week is broken into the following time patterns:

08:00 to 22:00	15 minute slots
22:00 to 08:00	1 hour slots

- Sign up for the 15 minute slots will start at 10:00 for the next day's time. Sunday and Monday start at 10:00 on Friday A maximum of four slots may be signed for. Please sign for only what you need.
- The overnight slots are signed for in two blocks. The Thursday, Friday, Saturday and Sunday overnight slots are available starting on Monday at 10:00. The Monday, Tuesday and Wednesday overnight slots are available starting on Friday at 10:00. In addition, there is the restriction that a user may only sign for ten 1 hour slots in any seven-day period.
- After 18:00 on all days, any time from 08:00 to 10:00 and 20:00 to 08:00 the next day may be signed for without restriction. The 'prime time' hours from 10:00 to 20:00 remain restricted until 2 hours before they start. Example: Slots from 13:00 to 13:45 become unrestricted at 11:00.
- Failure to login within the first five minutes of the first slot will result in cancellation of all contiguous slots. There are penalty fees charged to the faculty advisor if time slots are not canceled at least in advance or if the time slots are not used.

SIGN UP POLICY FOR THE Varian VNMRS-600 NMR INSTRUMENT
This is still being worked out. The time blocks are currently 30 minutes long and may be signed for up to 7 days in advance.

SIGN UP POLICY FOR THE Varian 400MR and 400SGM NMR INSTRUMENTS
There are no sign up for these instruments. All samples are queued and run in turn. The day queue is 60 minutes maximum per sample. All longer experiment sets will be shifted to the Night Queue which starts at 20:00.

Disk Policy for all Varian NMRs
NMR Storage: 145.0 GBytes Total Storage on each instrument
Roughly 30 Gbytes are reserved for the system. The rest is available for user use with no restrictions. All data files will be mirrored on NMRNET for 90 days after which it will be either deleted or sent to an archive location at the group advisor's decision. During this period you may use an FTP program on your computer to download the data. The parameters are:

Host: nmrnet.usc.edu
Protocol: SFTP
Logon type: Normal
User: 'Group name'
Password: 'Group password'

Varian Mercury 400
Data: nmrdata\username\400
Varian 400MR
Data: nmrdata\username\400MR
Varian 400SGM
Data: nmrdata\username\400SGM
Varian VNMRS-500
Data: nmrdata\username\501
Varian VNMRS-600
Data: nmrdata\username\600