New User Package for East Quad 600 NMR
Date: 01/25/08

Welcome to the East Quad NMR facility. This document contains information for new users using the 600MHz NMR at our facility. In this package, you will find:

1. Rules and Regulations
2. Basic instructions on NMR use
3. Information on the website
4. How to use the Scheduling calendar
5. Transferring your data

EQNMR Facility Rules of Use

The rules set forward in this document are intended to be fair to everyone using the NMR facility. Please feel free to discuss anything with the staff that you are concerned about.

1. Only authorized users will be permitted to use the spectrometer. Before a user can run any experiments, he or she must provide all required information to the facility staff. A New User form will be provided on the website for this purpose.

2. Users are expected to run their own samples. NMR facility staff will help as necessary to train users, but will not generally run samples. Non-standard experiments are the responsibility of the user.

3. All spectrometer use must be accounted for. When a user logs on to the spectrometer computer, time is automatically calculated until the user logs out. Failure to log out will result in overcharges.

4. <u>All instrument time must be scheduled using the on-line scheduling program.</u> Each lab will be provided with a username and password required to access the scheduling program.

5. <u>Users must show up for their scheduled time within 15 minutes of the starting time.</u> Failure to show up within this time may result in forfeiture of the time. Repeated failure may result in loss of spectrometer privileges.

6. <u>If a user cannot use their scheduled time, he or she must cancel their time **BEFORE** the start of their time block.</u> Users are expected to notify other users as soon as they are aware they will not be able to use their time. Charges will apply for use not cancelled prior to the starting time.

7. <u>If a user is not going to use all of his/her time block he or she must edit the online schedule to free up available time.</u> This has become such a major issue, that violations of this can no longer be tolerated. Every user gets three "strikes". The first violation a warning is sent to the user and his/her PI. Upon the second violation the users account is suspended for one week. A third violation results in the termination of the NMR account.

8. <u>Any requests which cannot be accommodated by the normal schedule must be submitted to facility staff at least one week in advance of the desired date.</u> The normal schedule will be interrupted to accommodate the request, after which time the usual schedule will continue. Every effort will be made to work with users to fulfill special requests.

9. <u>Users may sign up for their own time, but must follow the time limits.</u> A user may not sign up for more time than the maximum allowed slot during a given period, unless no one is scheduled after them.

10. <u>Experiments requiring a probe change require notification of the facility staff.</u> Only users authorized to change probes are exempted from this rule.

11. <u>**All safety regulations of the NMR facility must be followed at all times**</u>. If a user is unaware of the safety requirements, please consult with facility staff. If there are any further questions, contact the

Environmental Health and Safety Office. **Any emergency in the NMR lab should be handled by calling 911.** Failure to obey posted safety signs may result in personal injury or death.

12. No food or drinks may be brought into the NMR facility.

13. NMR samples must be properly labeled, and should be removed from the facility after the experiment is finished. Any samples left sitting around will be confiscated, and unlabeled samples will be disposed of promptly.

Basic instructions on NMR Use

Prior to using the NMR you must fill out a New User form (found on http://eqnmr.med.harvard.edu/ website) and have had a training session on the instrument.

The first step is to reserve time for your experiment. The following is the NMR time reservation policy:

*Short sign-up time***:** Mon-Fri 9AM-10AM and Sat 10AM-11AM. During this time, you can use the instrument for up to 15 min.

*Long experiment time***:** Mon-Fri 10AM-3PM and Sat 11AM-4PM. During this time, you can use the instrument for up to 2 hours.

Medium experiment time: Mon-Fri 3PM-7:30PM and Sat 4PM-7:30PM. During this time, you can use the instrument for up to 30 min.

Extra long block: Sun 10AM-7:30AM 5 hour max.

Overnight Runs: From 7:30 PM until 9 AM the next day, till 10 AM on weekends. This is primarily for long experiments, but if can be used for short ones if instrument is not being used.

*To sign up***:** go to http://eqnmr.med.harvard.edu/ and click on NMR Scheduling Calendar. Log in (lower right corner of the screen) using Laboratory user name and password. Select date and set up sign up time.

The following is a quick checklist of how to acquire a spectrum using the automated (walkup) account.

1. Log on: username _____, password _____
2. Insert your sample by clicking of **Eject** and **Insert** buttons and tune the probe if necessary (probe 1, channel 9) see picture of tune interface below

Note: Please make refrain from touching the lower portion of the spinner and white rings on the top while taking your sample in and out of the spinner. Extra dirt and oil on the spinner can clog the air jets in the instrument
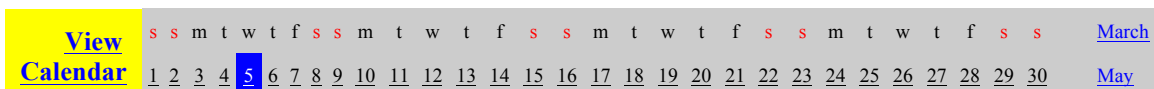
3. Be sure to select "New Automation Run" from the **File** option if this is your first time using the NMR that day. Choose the desired experiment (proton, carbon, homo or hetero 2D…)
4. If you want to use the automated locking and shimming, make sure the boxes for Auto locking(find z0) and Auto shimming(gradient shimming) are checked on the **Study, Start** tabs.
5. Double click yellow box that will appear on the lower left (time and exp).
6. Check solvent and acquisition parameters
7. Click **Submit** at the lower left
8. Wait for experiment to be completed
9. To save, under the **file** command there is a **Save as** option. To process: machine will automatically do it for you. Manual processing can be done with the menu side bar on the left of the display screen or can be done by clicking the **process** tab and selecting the appropriate menu.
10. Exit by clicking on **start**, then click **study** and then **eject** to take the sample out. Click **insert** to stop the air inside of the instrument.
11. On **File**, click **Exit Vnmrj.**
12. **Exit** the log in screen (The button can be found on the bottom bar)
12. Sign the logbook and indicate all the problems you noticed. If instrument is not working, please contact facility staff immediately[*]

The NMR Website

The website can be found on Harvard's intranet at http://eqnmr.med.harvard.edu/ It contains contact information for the staff, New User registration forms, a copy of the rules and regulations, and a link to the NMR scheduling calendar. It will also contain any important information and updates regarding the facility.

Please note that prior to using the NMR facility you MUST have filled out and sent in a new user registration form.

Using the Scheduling Calendar

This facility uses the program "Calicium" for scheduling time. The login for the scheduling calendar is in the lower right hand corner of the calendar. Your lab will be assigned a login name and a password that will allow you to modify the calendar. Once you are logged in, click on the date you wish to modify and you will be given the option to create an event. The figures below explain how to use the program:

View Calendar	s s m t w t f s s m t w t f s s m t w t f s s m t w t f s s	March
	1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30	May

NMR_Scheduling
April 5, 2006
Wednesday

Existing Events

9:00am - 1:00pm Open Access	Repeating Created by: Heffron	Edit Delete	Which instances: All ▼

Add New Event

Date: **When included in other calendars:**

April ▼	5 ▼	,	2006 ▼	Display this event ▼

Enter text for a new event:

Start Time: None ⊙ AM ○ PM

End Time: None ⊙ AM ○ PM

Category	Background	Foreground	Draw Border	
-	Default	Default	☐	Color Names

Enter a URL, or text for a popup window:

Anything starting with http:, https:, mailto:, ftp:, file:, or a 'www' string (e.g. *www.domainname.com*) will be a link. Anything else will be popup text.

Create Event   Reset

Repeat Information *(Optional)*

⊙ Don't Repeat

○ Repeat Every ▾ Day ▾   ☐ Skip Weekends

○ Repeat on the First ▾ **Wednesday** every month ▾

Repeat until ⊙ April ▾ 6 ▾ 2006 ▾ ,
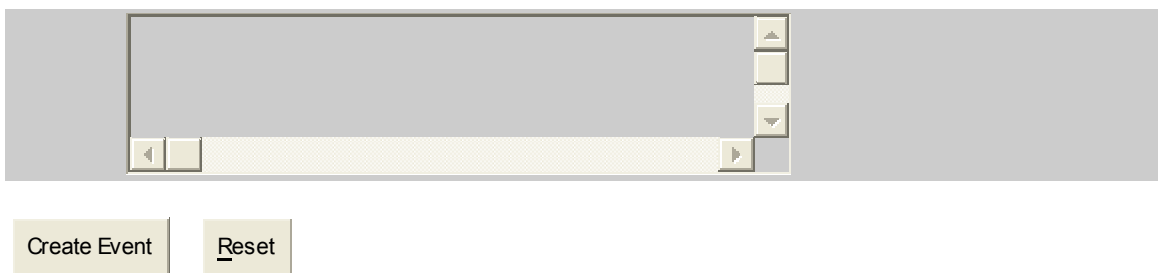
○ Forever!

Create Event   Reset

Email Notification *(Optional)*

Specify email addresses to notify that this event has been added. (Use commas between addresses.)

TO: _____   Email Address Selector

CC: _____

BCC: _____

Specify any additional comments that you would like included with the notification.

[Create Event]   [Reset]

In the "Add new event" section you will find a box to add text, usually your name, and you will be able to select the time you wish to reserve the spectrometer for. You can also add text, or an URL for a popup window. Once you are done simply click on create event to have your event added to the calendar. If you need to cancel your time, simply log back in and click the delete button next to your event. If your sample is taking longer than expected to prepare, and there is space open on the schedule, log in, click the edit button and change the time you wish to reserve

The NMR Workstation

When you submit a New User form we create an account for you on the NMR acquisition computer. This account stores your FID's and experimental parameters. However, while you are logged in to this machine, you are being charged for its use, even if all you are doing is analyzing data.

In the NMR room there is a third machine, not connected to a magnet. This is the NMR workstation. It has a newer version of software than the NMR acquisition machines. When you submit a New User form, we also create an account for you on this machine. Use of this machine will not be charged to your account. Therefore, if you have a long analysis of your data ahead of you, you may want to consider using the workstation. In the event of a heavy use period of the NMR, using the workstation for data analysis can really help you maximize your acquisition time.

Transferring your Data

There is one small hitch with using the workstation. There is not an easy straightforward method of transferring data from one machine to the other. In addition, there are several Labs who like to use third party software for analysis, who run into the same issue with transferring data from one machine to the next.

If you are familiar with UNIX, this is not such a big deal. If you are within the Harvard Medical School firewall, you can ssh and sftp to whichever machine you choose and transfer the FID. Since the fid is actually a directory file, do not forget to make a .tar file of the fid before transfer. The Names of the machines are nmr.med.harvard.edu for the acquisition machine and nmrwkstn.med.harvard.edu for the NMR workstation. If your lab frequently transfers files there should be someone who can show you the ins and outs.

If you are outside the University firewall you can log in to the NMR machine by logging on to http://secure.med.harvard.edu. using your ecommons username and password. Once there select the network connect option. This will download a gateway program to your machine that will allow you to tunnel through the university firewall. You should then be able to ftp your data to you own server.

Data Transferring Made Simple

Here is a simple set of instructions of one way to copy data from the NMR acquisition machine to the workstation. If you need help don't hesitate to ask one of the staff.

1) From the start up screen, once you have logged on to your account, open two file managers. You do this by clicking twice on the little icon that looks like a filing cabinet.

2) Both managers should open in your home directory where all your saved files are. On one manager go up three levels until you are in the root or "/" directory

3) In the root directory you will see a folder named "NMRdata". This folder is actually a link to the microbiology server. Open the folder. Inside you will find a folder labeled with your lab's name. Open this folder as well.

4) In the other file manager that still shows your home directory, select the file you wish to copy. Under the heading "selected" pick "copy to"

5) In the new window enter "/NMRdata/YourLabname" for the destination folder. This copies the data to the server. This is also a good way to back up your data. **NOTE:** Do not click and drag your file from one window to the other. This will permanently move it to the server and it will no longer be on the NMR machine.

6) Log on to the workstation, bring up the two file managers and go to your Lab's folder on NMRdata with one of them. Pick the file you wish to copy. Under "selected" pick "copy to" and pick "/export/home/ username" as the destination, where username is your username. You can now process your data on the workstation.

    Sometimes the NMR machine is in use and you don't have a chance to log in to transfer your files to the NMRdata server. There is another way to transfer your data to the workstation.

Slightly more complex data transfer

1) Select the file you want to copy to the workstation

2) Once you have logged in to your account on the workstation, right click the mouse. This will bring up a window.

3) Select Tools>terminal. A white window with a prompt "NMRWKSTN: username>" will appear

4)At the prompt type <u>exactly</u> the following (spaces included) substituting user with your username and filename with your filename

scp –r user@nmr:./filename ./filename

5) If it asks you to verify a key connection, type "yes" to proceed.

6) It will ask for your password at NMR:user, enter it.

7)Some text graphs will appear indicating the file transfer.

That's it! Your file is now in your account at the workstation.

Burning data CD's

You can also burn your data to a CD-R if you want a fast easy way to bring it out of the lab. It's also handy if you want to archive that crucial fid.

1) Log on to machine with the data you want to archive on it.

2) Insert a CD-R into the drive

3) Select Tools>terminal. A white window with a prompt "MACHINE: username>" will appear

4)Data from a Sun workstation has to be changed before it can be read by a PC or a Mac. What I am assuming here is that you want to archive your whole user directory and burn it to a CD. The next command copies your directory and turns it into a .iso file which can be read by a PC or Mac. Type at the prompt:

mkisofs –o filename.iso –R –s –hfs ./

5) the file "filename.iso' should appear in your home directory

6) type at the prompt:

cdrw –i filename.iso

7) The CD will burn and eject when it is done. You should then be able to read your data with any third party software on a PC or Mac.

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[*] Special thanks to Danica Galonic for allowing that her 600MHz spectrometer guide be used in this section.