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Howdy

Welcome to the Chemistry NMR Facility at Texas A&M University. Our facility includes 9 NMR spectrometers, an EPR, a helium recovery facility, and 3 full-time staff positions. Although this facility is physically housed within the Chemistry Department, we provide services to the entire TAMU community and beyond. More information about the facility and other topics of NMR interest can be found on our website (nmr.chem.tamu.edu).

Rules and Etiquette

There are some solid rules, and good practices.

- Never, for any reason, allow anyone else to use your login information. Violating this rule can have severe repercussions at the university level for all involved.
- Never, for any reason, are you to bring a person with a pacemaker close to a magnet.
- Avoid bringing ferromagnetic materials anywhere near a magnet. This includes most tools and office supplies.
- Be wary of bringing electronics close to a magnet, we are not responsible for their loss.
- Do not use an instrument for any purpose other than that for which it has been designed and set up.
- Do not make a reservation for anyone else or that you do not plan to keep, and delete a reservation if circumstances prevent you from keeping it as planned.
- Your experiment is expected to be completed within your reserved time.
- You may use an instrument without a reservation, but if someone has made a reservation during a time slot: that person has priority and you must surrender the instrument to that person.
- It is not, and never has been, anyone else's responsibility to save your data, deal with your sample, et cetera. You need to make arrangements beforehand if you cannot be present at the conclusion of your experiment, or you run the risk of losing it all.
- On systems with sample changers, never take someone else's sample out of the sample changer.
- Data should be saved in your data directory. We will not prevent you from saving data in other locations to which you have write privileges, but we will not backup data from any other location other than your data directory.
- When your immediate use for an instrument is done you should log out or change users. On systems where you log into the computer: log out. On systems where you log into the software: change users. Never power down a computer.
- Do not bring food or drink into the lab.
- A good rule of thumb is to not take actions that would disagree with you if someone else were to act similarly.

Faces

Faces is a web-based scheduling service that makes it possible for authorized clients to reserve resources on a first-come, first-served basis. Faces Scheduling System software is designed to facilitate the scheduling of scientific instruments and other resources in a research environment, and serves as a web accessible sign-up sheet. It was developed by William York and Saeed Roushanzamir at the Complex Carbohydrate Research Center at the University of Georgia.[1]

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[1] Taken from https://faces.ccrc.uga.edu/ccrcfaces/faq0.php

The NMR facility often makes use of Faces for reservations. Our group name is TAMU_CHEM_NMR. Your user name is usually your NetID, but there are circumstances in which we must provide you with a different user name. The Faces system does not authenticate through the university; in other words, your Faces password is independent of your NetID password. Please review our Faces Primer document for details about using Faces.

Working with CentOS Linux

The CentOS Linux distribution is a stable, predictable, manageable and reproducible platform derived from the sources of Red Hat Enterprise Linux (RHEL). The CentOS Project is a community-driven free software effort focused around the goal of providing a rich base platform for open source communities to build upon.[2]

Logging In

Our Varian systems are running CentOS 6, and are not using your NetID to login. However, your login name is still your NetID, as this is convenient for data and record management. You will set your password on each system separately, the NMR staff can reset your password but cannot tell you what it currently is. It is a violation to share your account with someone else, and you will face consequences for doing so.

Our other systems are running CentOS 7, and use your NetID to login. This means your login name is your NetID, and the password is your NetID password. Please remember that you must never share your password with anyone, there can be severe consequences. All password maintenance issues are handled by the IT Division (https://it.tamu.edu/services/accounts-and-id-management/uin-netid-and-passwords/netid/); in other words, we cannot help you should you forget your NetID password.

Logging Out

On systems where you login as a software user rather than a Linux user (eg: those walkup systems running in IconNMR like Tango and Waltz), do not log out the Linux user - ever. Use the "Change User" button in the software, only.

CentOS 6

To log out of a CentOS 6 machine look at the top of the screen and click on "System". Then scroll down to "Logout <NetID>", where <NetID> is your NetID, and click that. The screen will dim and a confirmation box will open, just confirm the logout and you're done.

CentOS 7

To log out of a CentOS 7 machine look at the top of the screen on the far right for a Power Button (⏻) and click it. From the dropdown menu then choose your name. From the options below your name choose logout. The screen will dim and a confirmation box will open, just confirm the logout and you're done.

Quotas and Notices

Upon logging in you might see a pop-up box. Do not ignore it. You need to read that pop-up and deal with the issue(s) it is notifying you of first; otherwise, things probably won't work out well. Usually the pop-up is notifying

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[2] Taken from https://www.centos.org/about/

you that you've exceeded the quota in your home directory, which is usually a result of improperly storing data there or web browsing. If you ignore the message and launch the software anyway, the software will just lock up and you'll have to have help getting it to work again. Which means you'll have to sit through the lecture about not ignoring the pop-ups. So, do us all a favor and deal with the issue(s) before launching the software.

Further Training

The NMR staff does not offer further training in using Linux, and you do not have to be extraordinarily proficient with Linux to use our systems. But the Laboratory for Molecular Simulation does hold a workshop that we recommend, as Linux is a very common operating system in academia and industry.

The Linux workshop is designed to teach a user the basic commands necessary to work in a Linux environment and consists of a 2 hour 45 minute lecture, and a 3 hour hands-on exercise session. This workshop is normally offered once during the Fall, Spring, and Summer sessions. The Linux workshop includes the following topics:[3]

- A brief introduction to the Linux operating system
- logging into a Linux machine
- basic Linux commands
- how to create and edit files (vi and nedit)
- how to compress/store files/directories (gzip and tar)
- how to set up configuration files (.bashrc, .tcshrc, .cshrc, etc)
- searching file content (grep)
- regular expressions (wildcards)
- input/output redirection
- remote logins (ssh)
- transferring files between two Linux machines (sftp/scp)

Working with VnmrJ 4.2A

First Steps

Start VnmrJ 4.2A by double-clicking on its desktop icon. The program loads with a splash screen on top that shows the names of the fields within the software. There is a little check box in the lower left that will disable this feature for future starts, we suggest you toggle that check box. Next there is a "X" box in the upper right that will dismiss the splash screen. You are now ready to use the system, start by tapping the experiment you want to run in the Experiment Selector.

Don't Panic. If you're a seasoned VnmrJ user, (almost) all of your old commands, macros, et cetera will work fine in this version of VnmrJ. You do not *have* to change your ways, but things are sometimes in different places, work a little differently, or have better options than they did in days of old. The rest of this guide is going to focus on using the GUI, but if you prefer the command line feel free to use it instead.

With the Start tab selected, click the Eject button. Take the placeholder sample from the magnet's upper barrel, remove the tube from the spinner, and carefully install your tube into the spinner – make sure to use the depth gauge to align it properly. Then place your sample in the upper barrel, go back to the software and click the Insert

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[3] Taken from the LMS website (https://lms.chem.tamu.edu/).

button. The gas flow supporting the sample should cease, and soon you will hear a small thunk, which indicates the sample has reached the bottom of the upper barrel.

Next you need to set the lock phase, which means locking by hand. You should only have to do the locking by hand once in a long while, but every now-and-then the lock phase does change. To lock a system by hand go through the following steps:

1. In the Start tab's Sample Info page: choose the correct solvent
2. Choose the Start tab's Lock page and start the Lock Scan by tapping its button.
3. Make sure the check box next to Lock is empty.
4. Manipulate z0, lock power, lock gain, and lock phase until an appropriate profile is produced.
5. Toggle the checkbox next to Lock.
6. Turn off the Lock Scan by tapping its button.

The lock phase does vary some from sample-to-sample, but not very much. Truth is, close counts. In fact, there is an entry for the lock phase in the probe file, so you might want to start there. However, sometimes, usually following a power outage, the lock phase shifts by some random increment of 90-degrees. If in later use the lock level is very low (lock profile incorrect), this sort of shift may be the root cause – so it's good to know how to manually lock. But from here on out, barring a lock shift, you should be able to just take the Auto Lock button in the Start tab to do all your locking.

Direct Acquisition

This is the type of acquisition that seasoned VnmrJ users are used to. There are several steps summarized here, which will be detailed more in training. To acquire a spectrum using the direct method use the following steps:

1. Put your sample in the magnet.
2. Click the base experiment you wish to run in the Experiment Selector. The experiment should load and display the pulse sequence's rf profile.
3. In the Start tab's Sample Info page: choose the solvent.
4. In the Start tab click the Auto Lock button, and wait for it to complete. If this should fail, try locking by hand (see above).
5. In the Start tab click the Gradient Shim button. The spinning should stop for gradient shimming, you should not be spinning when gradient shimming.
6. You can start the spinning up from the Start tab's Spin/Temp page by hand if you want to (choose 20Hz). Once the spinning is stable you may polish the z-shims by hand if you like. In general, this step is unnecessary, as the spinning will start automatically later and the shims are often good enough following gradient shimming.
7. On some systems you should tune the probe. On systems that require tuning, you may do the tuning any time after your sample is in the magnet and before you start your acquisition (assuming the temperature isn't being changed).
8. Next, in the Acquire tab customize the pulse sequence using the fields on the pages within the Acquire tab. You'll find entries for every parameter used by your chosen experiment within these pages. Often a given parameter will appear in more than one location, as a convenience.
9. When everything is ready (including making sure the experiment ends at an appropriate time) then tap the Go button. Sit back and wait for a block to complete, then either sit some more or process the data acquired to that point.

10. Once the system has become idle again, go to the File menu, mouse down to Save As and **save your data in your data directory**. Your data directory is within the vnmrsys directory and called data (~/vnmrsys/data), and is actually a link to your data drive location on the system.
11. Start a new sample, or replace the placeholder. There's no need to lock the placeholder or anything, just please put it back – don't leave the magnet empty.

Study Queue Acquisition

The Study Queue can be really handy for setting multiple things up, and letting the software do more work for you. You still have to get the lock phase (roughly) correct, so do not fail to get your first lock on the system by hand. The Study Queue lives in the bottom left corner of the software interface, to acquire data using the Study Queue do the following:

1. Put your sample in the magnet.
2. If you need to tune the probe, do so before working with the Study Queue.
3. In the Study Queue area, click New Study.
4. In the Start tab's Sample Info page choose the solvent, provide a sample name, and choose any options there you fancy. It is recommended that you provide some comments, at least.
5. Choose the experiment(s) from the Experiment Selector. Notice that they are added to the Study Queue in order. If you have added one in error, just drag-and-drop it to the trashcan in the bottom left.
6. Double-click each experiment and customize it within the Acquire tab's pages.
7. When all is ready, click submit. The system should now lock, shim, and acquire all the experiments in the queue. It will also *automatically* save the data in your data directory using the sample name, date, and a counter.
8. Start a new sample, or replace the placeholder. There's no need to lock the placeholder or anything, just please put it back – don't leave the magnet empty.

Normally, that's it. Yes, you can stop a queue and do all sorts of fancier stuff, but that will be covered further in training. One thing to mention here at least would be if you forgot to run an experiment on a sample, you can append that experiment by choosing to Continue a queue rather than start a new one. Oh, and the queue will show you the time required to complete each experiment and the queue as whole – no excuses for going over time.

Processing with Workstations

Local Data

As previously mentioned, your data should be saved in your ~/vnmrsys/data directory, which is a local directory. (That means the files are stored on a hard drive in the computer you used to collect the data). In most cases, you may retrieve your data from the local hosts by using sftp or by using a flash drive, but this is NOT true for instruments being run in walkup/Icon-NMR.

System Identification

The NMR facility has two workstations in 1318. These workstations run TopSpin, and have <u>all of your data</u> independently backed up on them. These systems are:

1. Proton
    (proton.chem.tamu.edu, 165.91.176.232)

2. Deuterium
    (deuterium.chem.tamu.edu, 165.91.176.225)

Data backups are automatically done periodically, and the system backups are offset. There are also scripts running that copy over your data when you login. (It is the act of logging in that triggers these scripts, remaining logged in does not trigger them again).

On these systems your data may be found in a directory with your adviser's name, your login name, and the instrument name. For example, if kjhwang from the Payne lab had acquired her data on the VnmrS 500MHz, her data would be in /data/payne/kjhwang/nmrs500. There is a link to /data on your desktop, and you can browse to the appropriate location from there.

You may retrieve your data from these workstations by using sftp or by using a flash drive.

TopSpin

TopSpin is software from Bruker that may be downloaded for free for academic purposes. If may be found here: https://www.bruker.com/service/support-upgrades/software-downloads/nmr/free-topspin-processing/free-topspin-download.html. We suggest assigning your data directory to your login named directory (/data/payne/kjhwang in the above example), and then tunneling into the sub-directories to import/convert your data from there.

Appendix

Not every system is capable of running every experiment. Please check the NMR facility's webpage (nmr.chem.tamu.edu) for more details on each system; and, if still in doubt, please ask the staff for clarification.

Variable Temperature

Training for using each system at temperatures away from room temperature are separate classes. You are not allowed to to perform studies that require temperature control without first having taken the class for the specific instrument on which you intend to run them. Variable temperature studies often take a considerable amount of time, and are generally run in off-peak times (nights and weekends).

(CIL) Solvent Chart

Solvent	^{1}H δ (ppm)	JHD (Hz)	^{13}C δ (ppm)	JCD (Hz)	HOD ^{1}H δ (ppm)	ρ (g/mL)	MP (°C)	BP (°C)	κ	gmw (g/mol)
Acetic Acid-d4	11.65 (1) 2.04 (5)	2.2	178.99 (1) 20.0 (7)	2.0	11.5	1.12	17	118	6.1	64.08
Acetone-d6	2.05 (5)	2.2	206.68 (13) 29.92 (7)	0.9 19.4	2.8	0.87	-94	57	20.7	64.12
Acetonitrile-d3	1.94(5)	2.5	118.69 (1) 1.39 (7)	21	2.1	0.84	-45	82	37.5	44.07
Benzene-d6	7.16(1)		128.39 (3)	24.3	0.4	0.95	5	80	2.3	84.15
Chloroform-d	7.27 (1)		77.23 (3)	32.0	1.5	1.50	-64	62	4.8	120.38
Cyclohexane-d12	1.38 (1)		26.43 (5)	19	0.8	0.89	6	81	2.0	96.24
Deuterium Oxide	4.80 (DSS) 4.81 (TSP)		NA	NA	4.8	1.11	3.8	101.4	78.5	20.03
N, N-Dimethyl-foramide-d7	8.03 (1) 2.92 (5) 2.75 (5)	1.9 1.9	163.15 (3) 34.89 (7) 29.76 (7)	29.4 21.0 21.1	3.5	1.04	-61	153	36.7	80.14
Dimethyl Sulfoxide-d6	2.50 (5)	1.9	39.51 (7)	21.0	3.3	1.18	18	189	46.7	84.17
1,4-Dioxane-d8	3.53 (m)		66.66 (5)	21.9	2.4	1.13	12	101	2.2	96.16
Ethanol-d6	5.29 (1) 3.56 (1) 1.11 (m)		56.96 (5) 17.31 (7)	22 19	5.3	0.89	<-130	79	24.5	52.11
Methanol-d4	4.87 (1) 3.31 (5)	1.7	49.15 (7)	21.4	4.9	0.89	-98	65	32.7	36.07
Methylene Chloride-d2	5.32 (3)	1.1	54.00 (5)	27.2	1.5	1.35	-95	40		86.95
Pyridine-d5	8.74 (1) 7.58 (1) 7.22 (1)		150.35 (3) 135.91 (3) 123.87 (5)	27.5 24.5 25	5	1.05	-42	116	12.4	84.13
Tetrahydrofuran-d8	3.58 (1) 1.73 (1)		67.57 (5) 25.37 (1)	22.2 20.2	2.4-2.5	0.99	-109	66	7.6	80.16
Toluene-d8	7.09 (m) 7.00 (1) 6.98 (5) 2.09 (5)	2.3	137.86 (1) 129.24 (3) 128.33 (3) 125.49 (3) 20.4 (7)	23 24 24 19	0.4	0.94	-95	111	2.4	100.19
Trifluoroacetic Acid-d	11.50 (1)		164.2 (4) 116.6 (4)		11.5	1.50	-15	72		115.03
Trifluoroethanol-d3	5.02 (1) 3.88 (4x3)	2 (9)	126.3 (4) 61.5 (4x5)	22	5	1.45	-44	75		103.06