Acquiring Data in VnmrJ 4.2A

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Linux Primer

Initial Steps

You have been given a login name and temporary password. Provide these to the system at the login screen. The system should then inform you that you must change your password right away, and will ask for the following information in order:

1.Your temporary password (the one you were given)

2.Your new password (the one replacing your temporary password, choose one that is less than 14 characters long)

3.Your new password again (as a check to be sure you typed it correctly the first time)

From then on your password **on that system** will be the new password you provided above, unless you change it again. Your password for that system on the reservation system will update later that evening.

At this point you should be logged in and will soon see your desktop. At the top of the screen is a **System** menu, click on it. Roll the mouse down the menu to **Preferences**. Next roll the mouse over to the Preferences sub-menu and down to **Screensaver**, and click on it. In the Screensaver popup, un-check the box next to "Lock screen when screensaver is active". The close the Screensaver popup.

Reservations

Reservation rules vary from system-to-system, but may be found here: http://nmr.tamu.edu/rules.php. The reservations are handled by nmrsun1 (nmrsun1.chem.tamu.edu, 165.91.176.180) using a reserve program. Use ssh to login to nmrsun1 with your login name and current password (which, again will start off as the temporary password you have been given). The system will rattle off a motd (message of the day) that always includes a reminder of proper usage, and the penalties for failing to do so. The system will then prompt you to "Input display server name:" - just hit enter. You should now see a prompt appearing as "ID 1>", where ID is your login name. At this prompt type "su reserve" and the system will prompt you for your terminal type, if you're unsure choose "1: DEC vt100 (default)" by keying in "1" followed by a return. You should now see a listing of all the systems.

Hit the number key for the system you'd like to reserve. A new motd for that system may flash up, please read that message as this is where the NMR staff will inform you of any special conditions affecting the system. After you dismiss the system's motd with a keystroke, you will be prompted for your "Instrument User Name". If you leave the "Instrument User Name" field empty you may browse the system's current reservations, but may not make one yourself – you may do this even if you do not have an account on the chosen system. However, to actually make a reservation you must supply the login name you have been provided as the "Instrument User Name" and hit enter. You will then be prompted to provide the password for that instrument – yes, you could set up each instrument with its own password if you like.

Once you have logged into the system's reservation, you will be presented with a user information form. The only required fields are phone number (in format #-####, including the dash) and lab/room

number, but we suggest you fill out the rest of the information as well. When you are done, press "D" and you will be taken to the actual time reservation screen. Permitted operations are located at the bottom of the screen, and the system automatically enforces the reservation rules. When you are done making/viewing your reservation, press "Q" to quit and return to the full list of systems. You may then choose another system, or exit the reservation program by hitting "Q". Once you have left the reservation program, logout of nmrsun by typing "logout" at the command prompt – do not leave the system logged in while not in immediate use.

Reservation Terminals

In many of the NMR labs there is a second computer that is only for making/viewing reservations on that system. This computer should already be logged into the appropriate Linux account, but the reservation system may not be running on it. To get the reservation system running double-click on the "Start reserve.sh" icon and ask it to "Run in Terminal". Then wait, it takes a minute. Soon though, you will be asked the same questions you would be presented if you had logged into nmrsun1. Tell the program that it's a VT100 terminal, choose the system, et cetera.

Rules and Proper 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 make a reservation you do not plan to keep, and delete a reservation if circumstances prevent you from keeping it as planned.

•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.

•Do not use an instrument for any purpose other than that for which it has been designed and setup.

•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.

•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, log out. Do not switch user. Do not let the window sit there open. Log out.

System Identification

The TAMU chemistry department systems are detailed below. The name of the computer associated with the system and its IP are included as well. Please note that each system is described for the

configuration you are most likely to find, but other options may be utilized – check first.

1.VnmrS 300MHz System in Room 2211E

    (nmrs300.chem.tamu.edu, 165.91.176.184)

    This system is setup solely for the organic labs. It is running VnmrJ 4.2A software in a Walkup mode – where operators do not log into the Linux account, but into the software.

2.Mercury 300MHz H/C System in Room 2413

    (mercury300.chem.tamu.edu, 165.91.176.183)

    The installed probe is tuned solely to 1H and 13C, so the system should not be used for any other nuclei. Training on this instrument is part of your initial introduction to NMR.

3.Inova 300MHz H/F/P/C System in Room 313

    (inova300.chem.tamu.edu, 165.91.176.182)

    The current probe does not have a gradient coil, which limits your experiment choices. However, the system is certainly capable of running routine 1D H/F/P/C spectra. Training on this instrument is part of your initial introduction to NMR.

4.Inova 400MHz Broadband System in Room 1324

    (inova400.chem.tamu.edu, 165.91.176.68)

    This instrument has been designated for running nuclei other than H/F/P/C, and is equipped with a H/F{X} probe. It is capable of running H/F/P/C as well, but should someone require a nucleus outside these four – that person will have priority on this instrument.

5.Inova 500MHz H/F/P/C System in Room 1221

    (inova500.chem.tamu.edu, 165.91.176.67)

    This system has a quad probe allowing it to do a wide-range of H/F/P/C experiments. Any choice of one high-band (H/F) and one low-band (P/C) nuclei may be done on this system.

6.VnmrS 500MHz H/F/P/C System in Reed-McDonald Room 415

    (nmrs500.chem.tamu.edu, 165.91.223.23)

    This system has four channels, and a matching probe. This means that any combination of H/F/P/C experiment may be done on this system.

7.Avance III 400MHz Broadband System in ILSB Room 1163

    (ilsb400.chem.tamu.edu, 128.194.148.6)

    This system is capable of running proton with (almost) any choice of second nucleus. The system is equipped with a 16 position sample changer and automatic tuning.

8.Avance 500MHz Cryoprobe System in Room 1238

    (avance500.chem.tamu.edu, 165.91.176.142)

    This system is equipped with a H/C/N cryoprobe, and is meant for long acquisitions of low concentration samples. It is equipped with a 24 position sample changer and automatic tuning.

9.VnmrS 500MHz Broadband System in Room 1327

    (nmrs500.chem.tamu.edu, 165.91.176.178)

This system is equipped with a H/F{X} probe, and is similar in role to the Inova 400MHz above. This system has been set aside for very long acquisitions.

10. Avance 400MHz Solid-State System in Room 1321

(avance400.chem.tamu.edu, 165.91.176.66)

This system is setup to run solid-state experiments only. It is not on the reservation system, contact Vladimir Bakhmoutov (1323 Chemistry, 979-845-8993, bakhmoutov@mail.chem.tamu.edu) to schedule time.

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.

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 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 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 check box 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. (Skip this step and shim by hand if the probe cannot gradient shim – like the one on the Inova 300MHz). 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. 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.

8. 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.

9. 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).

10. 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

This is new to VnmrJ for most people. It 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. In the Study Queue area, click New Study.

3. 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.

4.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.

5.Double-click each experiment and customize it within the Acquire tab's pages.

6.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 he sample name, date, and a counter.

7.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 require 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 the hard drive of the computer you used to collect the data). You may retrieve your data from the local hosts by using sftp (see above for system names and IPs) or by using a flash drive.

System Identification

The NMR facility has also set up two workstations in 1318. These workstations run TopSpin, and have all of your data 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 currently done every half hour on both systems, and the systems are offset by a quarter hour. There are also scripts running that copy over your data on demand, but if you wait no more than half an hour one of these workstations will have your data available.

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.