Acquiring Data in OpenVJ

Howdy

- Rules and Etiquette
- System Introduction and Identification
- Faces Reservations
- Training Flow Chart

Working with Alma Linux

- Logging In
- Logging Out
- Further Training

Working with OpenVJ

- First Steps
- Direct Acquisition
- Study Queue Acquisition

Processing with Workstations

- Local Data
- System Identification
- Processing Software
    - TopSpin
    - MNova

Howdy

Welcome to the Chemistry NMR Facility at Texas A&M University. Our facility includes eight NMR spectrometers, an EPR, and three full-time staff positions. Although this facility is physically housed within the Chemistry Department, we provide services to the entire TAMU community and beyond.

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 make a reservation you do not plan to keep, and adjust a reservation if circumstances prevent you from using it as planned.
- On systems with sample changers, never take someone else's sample out of the sample changer.
- 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 set up.
- The NMR staff often posts notices in the instrument rooms, please read them and deal with them as necessary.
- 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 or change user. Do not switch user. Do not let the window sit there open. Do not power down.

System Introduction and 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. Avance Neo 400 MHz Broadband Systems in Room 1318 (Tango and Waltz)
   These systems are capable of running proton with (almost) any choice of second nucleus. The systems are equipped with a sixty position sample changer and automatic tuning. They run in Icon-NMR as first-come, first-served and so are not on the reservation system. All initial NMR training is done on these systems by taking a two hour class that is held every Tuesday morning at 0900 hrs in room 1318.

2. Avance Neo 500 MHz Broadband System in Room 1321 (Salsa)
   This system is intended for long term studies, including variable temperature (VT) studies. Training is scheduled on demand for up to four eligible candidates per session. Eligibility is based on usage on Tango and Waltz. VT training is separate and users must be comfortable with running the system manually.

3. Avance 500 MHz Cryoprobe System in Room 1238 (avance500)
   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 twenty four position sample changer and automatic tuning. Training for this system must be scheduled with the staff, initial training is done for using the system under Icon-NMR only.

4. VnmrS 500 MHz Broadband System in Room 1327 (nmrs500)
   This system has several probes, but usually has a quad probe installed that allows it to do a wide-range of H/F/P/C experiments. In this configuration, any choice of ^{1}H or ^{19}F and ^{31}P or ^{13}C may be acquired. This is a reserved system and occasionally large blocks will be assigned for those requiring other probes.

5. VnmrS 500 MHz H/F/P/C System in Reed-McDonald Room 415 (reed500)
   This system has four channels, and a matching probe. This means an experiment with any combination of H/F/P/C may be done on this system. Training for this system is available to users that have completed training and shown usage on nmrs500. Contact the staff to arrange training.

6. Avance III 400 MHz Broadband System in ILSB Room 1163 (ilsb400)
   This system is capable of running proton with (almost) any choice of second nucleus. The system is equipped like Tango and Waltz, and runs in Icon-NMR as first-come, first-served and so is not on the reservation system. This system is only available to ILSB users, but training for this system is done every Tuesday morning at 0900 hrs in the Chemistry Department room 1318.

7. Avance 400 MHz Solid-State System in Room 1324 (Foxtrot)
   This system only runs solid-state experiments. Training on this system is more of an apprenticeship, so if you are going to be a significant, consistent user contact Vladimir.

Faces Reservations

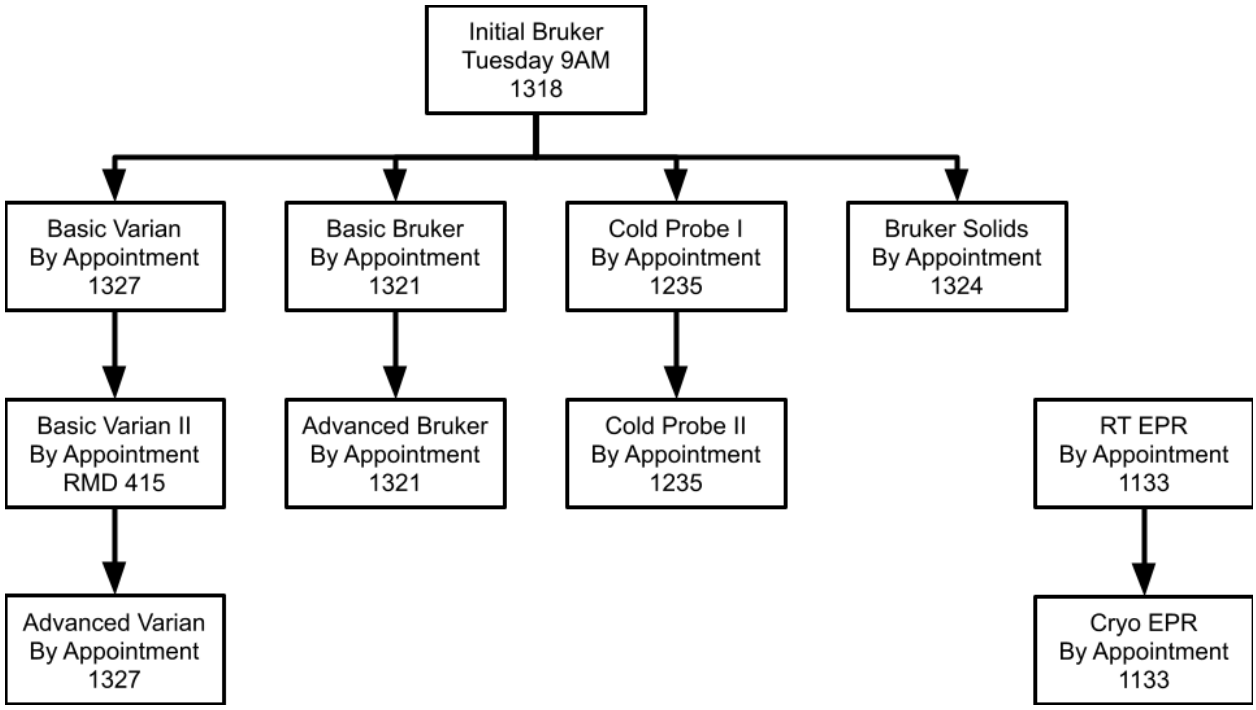
Faces is a web-based scheduling service that makes it possible for authorized clients to reserve resources on a first-come, first-served basis. Faces Scheduling System software is designed to facilitate the scheduling of scientific instruments and other resources in a research environment, and serves 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.

The Faces website is https://faces.ccrc.uga.edu and our group is TAMU_CHEM_NMR. You will be sent an email with your user name and initial password. Please see the Faces Primer document in our Training Center (https://nmr.chem.tamu.edu/TrainingCenter.php) for more details, including specific time allotments and rules for each

system. All NMR instruments that require a reservation are scheduled through Faces, but you will only see those systems for which you have been trained.

Training Flowchart

Training on our systems is done in an order that adds incrementally to the understanding required to use the system correctly, and is shown in the flowchart below



Variable temperature training for each system (if appropriate) is a separate training on that system and is arranged by appointment.

Working with Alma Linux

An Open Source, community owned and governed, forever-free enterprise Linux distribution, focused on long-term stability, providing a robust production-grade platform. AlmaLinux OS is binary compatible with RHEL®.[1]

Logging In

All of our systems use your NetID to login, regardless of whether you are logging into the computer or the software. This means your login name is your NetID (not your UIN and without the @tamu.edu), and the password is your NetID password. Please remember that you must never share your password with anyone, there can be severe consequences for all involved. 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.

[1] Taken from https://almalinux.org/

Logging Out

On systems where you login as a software user rather than a Linux user (eg: those walkup systems running in Icon-NMR like Tango and Waltz), do not log out the Linux user - ever. Use the “Change User” (👤) button in the software, only. Never power down any facility computer without contacting the NMR staff first.

To log out of an Alma 9 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 “Power Off/Log Out”. From the options in that dropdown menu choose “Log Out”. The screen will dim and a confirmation box will open, just confirm the logout and you’re done. Again, never power down any facility computer without contacting the NMR staff first.

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 two hour forty five minute lecture, and a three 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:[2]

- 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)

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

First Steps

Start OpenVJ 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 OpenVJ. 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 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. (Skip this step and shim by hand if the probe cannot gradient shim). 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 20 Hz). 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

This is new 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. 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. (Meaning the files are stored on a hard drive in the computer used to collect the data). You may retrieve your data using a flash drive, but it must be formatted in FAT.

System Identification

The NMR facility also has two workstations in 1318. These workstations run have all of your data from every instrument backed up on them. These systems are:

1. Proton
   (proton.chem.tamu.edu, 10.119.120.25)

2. Deuterium
   (deuterium.chem.tamu.edu, 10.119.120.26)

Data backups are done very frequently, so you should not have to wait long for your data to be available on these servers.

On these systems your data may be found in a directory with your advisor'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.

You may retrieve your data from these workstations by using sftp or by using a FAT formatted 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.

MNova

MNova is a powerful, popular software from Mestrelab Research (https://mestrelab.com/) for which TAMU has a campus-wide license. Licenses and software may be downloaded from the TAMU IT software store (https://software.tamu.edu/) it is free for your use at TAMU.

(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