General Information for Automated Inova 500 MHz NMR (Indy) and Mercury Plus 300 NMR (Hg3)

Primary Contacts:
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Hg3 GLA: Haoxuan Wang, x6061, hxwang@caltech.edu

Indy is capable of running ^{1}H and ^{13}C spectra; it costs $20/hour. Hg3 does ^{1}H, ^{13}C, ^{19}F, and ^{31}P; it costs $15/hour. Other nuclei do not work on these instruments as currently configured and should not be attempted. The automated 400 instrument Siena and the manual 500 Daytona are both fully multinuclear. Note that Hg3 has much lower ^{13}C sensitivity than Indy, and long experiments on Hg3 are generally not cost effective by comparison.

Preparing for your experiment

Here are a couple of things to do before setting up your NMR experiment:

1. Subscribe to the NMR users mailing list. You can do so at this website: https://utils.its.caltech.edu/mailman/listinfo/nmrusers
2. For the night queue only, you must sign up for time on WebCal. The website is: http://chemex.che.caltech.edu/cgi-bin/webcal-3/webcal.cgi?function=webday&cal=Indy (for Hg3, substitute hg_3 at the end)
3. Before using the spectrometer, you must **ALWAYS** sign the log book. Enter the **date, your initials and group initials, your lab extension** (or a number at which you can be reached if you are an off campus user), **sign up time, nuclei and solvent**

Sample Preparation:

All NMR tubes must be ***8 inches in length, 5 mm in diameter***. The autosampler will likely fail (quite possibly breaking your sample) with tubes that are taller or shorter. Don't use any tubes that are chipped or cracked at the top—they are prone to being crushed by the gripper fingers. Samples should contain 5 cm of solvent to ensure optimal autolocking and autoshimming. Additional solvent won't help anything and just wastes money.

Sign Up Rules:

		Indy	Hg3	
Every day	8 am – 8 pm	1 hr		Day Queue
Every evening	8 pm – 11 pm	2 hr		Day Queue
Every night	11 pm – 8 am	9 hr		Night Queue
Every day	8 am – 12 mid		30 min	Day Queue
Every night	12 mid – 8 am		8hr	Night Queue

You may use e.g. the 1 hour daytime limit on Indy to run more than one sample back to back, as long as what you are submitting at one time does not *total* more than one hour. If you use one hour during the day, and then submit more samples, there should be a minimum of one hour in between those submissions.

Login:

On the login screen, type or select your username from the *Operator* drop down menu. Enter your password. Click *OK*.

Sample Loading:

Before loading your NMR tube into the rack, check the sample tray menu in VnmrJ to see which slots are available. They are color-coded:

Gray	Available
Green	Completed
Blue	Active
Yellow	Submitted – Day Queue
Purple	Submitted – Night Queue
Red	Error
Faded / Greyed out	Sample submitted by an operator other than the one currently logged in

Load your sample into a spinner, adjust its height using the depth gauge, wipe the bottom with a kimwipe, and load it into an open slot.

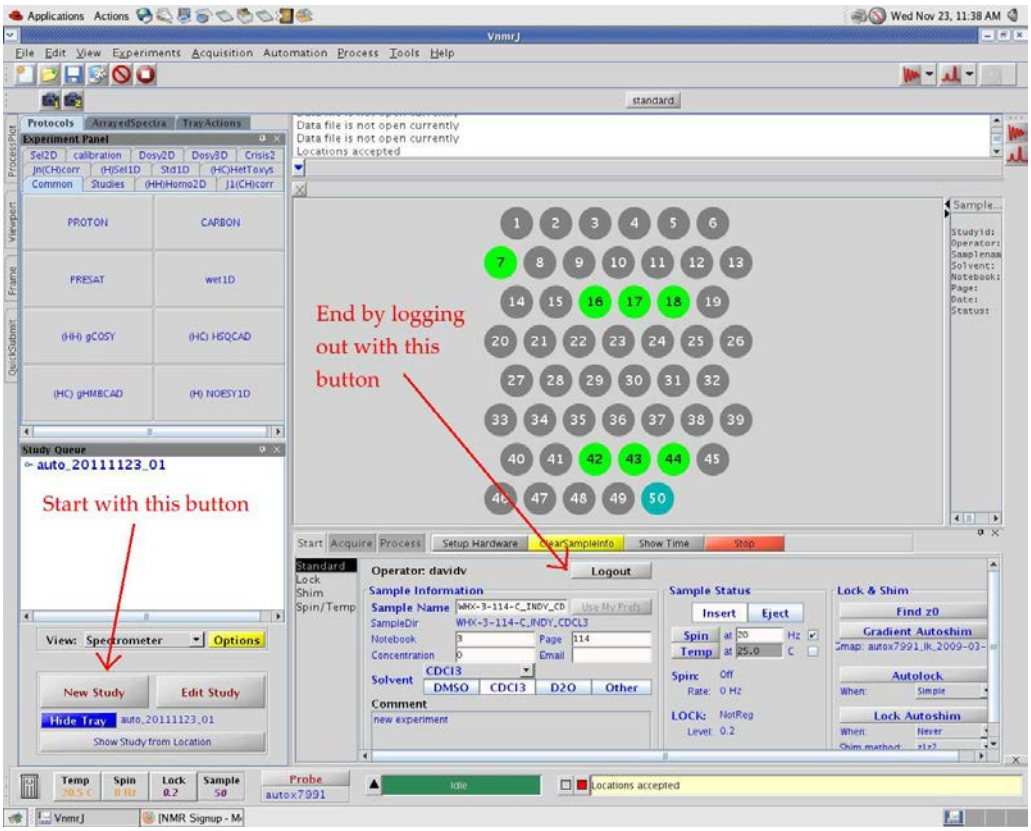
Please leave the spinners in the ***large white test tube rack*** when not in use. If there are no available spinners, remove an NMR tube from a slot that has already been run, place it in the ***small green test tube rack*** next to the autosampler tray, and use that spinner.

Do NOT submit to slot #50. This is reserved for the standard.

If the autosampler malfunctions, it can be turned off by pressing the RED button. If you press the red button, you must call a facility administrator or the GLA to reset the instrument. If you cannot find an administrator or GLA, leave a note on the instrument and send us an email.

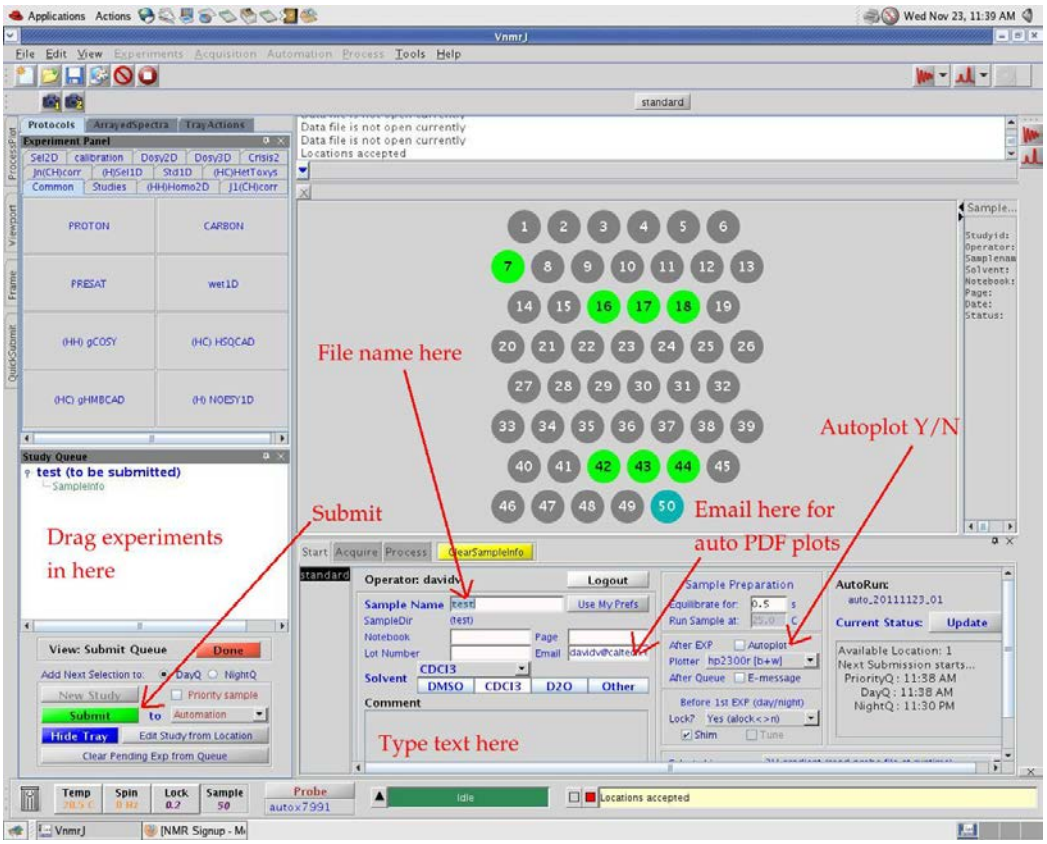
Sample Submission:

This software has two operating modes: study management and submit modes. You will start in the study management mode, which looks like this:



Start by clicking the “New Study” button on the bottom left of the screen. This will add a new study to the “Study Queue” panel on the left side of the screen. On the “Start” panel, enter a sample name for your sample. What you enter in the “Sample Name” field will become the name of your saved data folder. You MUST enter something in this field! On the same panel, select your solvent and enter an optional comment that will appear on your spectrum.

Once you have entered all the required information in the study management page, select your desired experiments from the “Experiment Tabs” on the left side of the screen. It will look as shown below:



To modify an experiment, double-click on the title of the experiment in the "Study Queue." The experiment that is currently being modified will be in a bold, italicized font. From the "Acquire" tab, select the parameters for your experiment. Once you have modified each of your experiments as you please, click the "Show Time" button to update the total experiment time in the study queue. (You will need to note this in the logbook.) Click the "Show Tray" button below the study queue, and select the position of your sample in the black sample tray. If properly selected, the position will show a multicolor, rotating border. Be sure to check either the "Night Queue" or "Day Queue" box appropriately. Finally, click "Submit" followed by "Done."

At any given time, you can edit studies or delete ones that have not yet run. Click on the Edit Study button to begin; you may add experiments, modify experiments, or cancel experiments by dragging them to the trash can. After you make your changes, click the green "Submit Button" again.

All entries must be recorded in the logbook.

Autosaved Data:

All data is automatically saved in a folder that corresponds to the "Sample Name" entered in the sample management mode. You can find your data in the following location:

`/home/operator (you)/vnmrsys/data/SampleName`

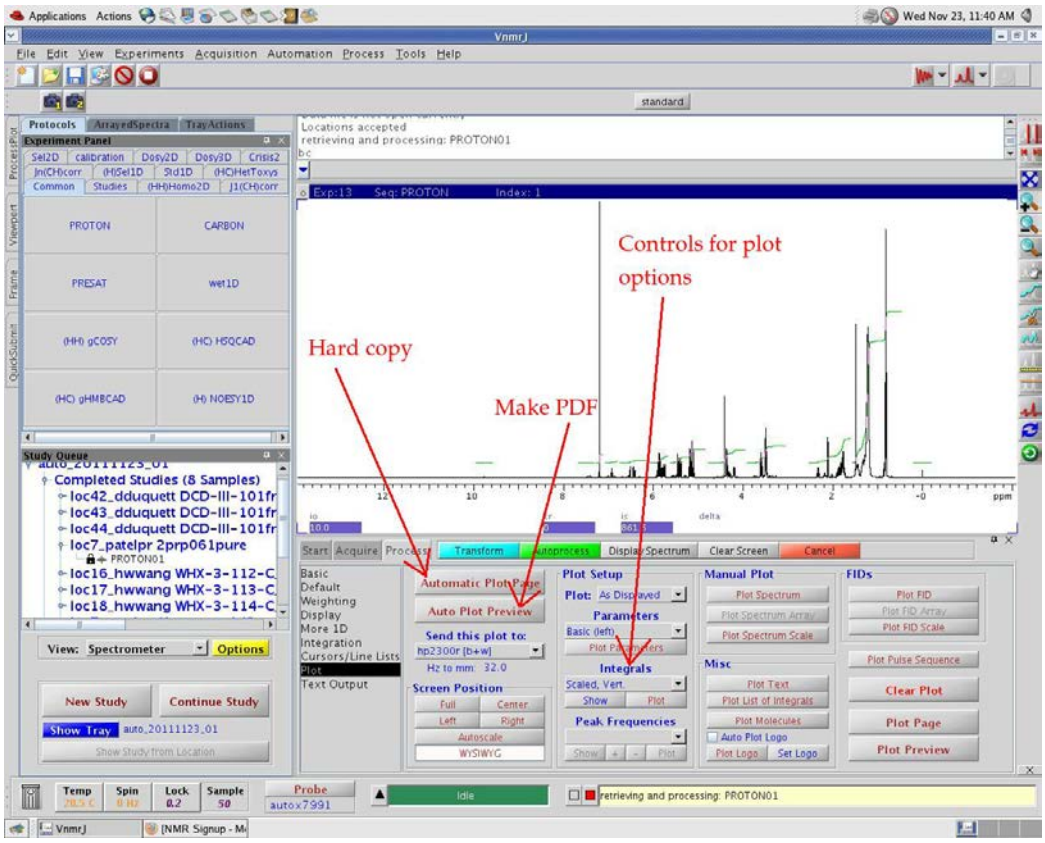
Within the folder for a given sample will be additional subfolders (with names like Proton01, Carbon01, etc.) for each experiment that contain the raw data within them.

If you entered an e-mail address in the sample management window as well, you will receive an e-mail with a pdf file of your spectrum following acquisition.

Data Processing:

You do not need to resave your data under another name. It is automatically backed up nightly to the NMR server, but it is an excellent idea to make your own backup periodically!

All old processing/plotting commands still work. However the buttons on the Process -> Plot tab may be more convenient to use than the command line.



A typical protocol for replotting a spectrum with custom integrals might be:
Cut the integrals as you wish them to be.
Type the command “bc” to flatten the baseline, after which integral level and tilt adjustments are seldom needed.
Then click either the Automatic Plot Page or Auto Plot preview buttons. The preview function creates a PDF file which you can save, email to yourself, or plot.
You can retrieve your data with a USB stick or directly from the instrument or the server, using a network drive. Instructions for accessing the network drives can be found at http://mangia.caltech.edu/NMRdata.html.

Many groups choose to process their spectra on MestReNova. Instructions for downloading this program can be found at http://mangia.caltech.edu/mestrenova.html

All sorts of other useful information can be found on the NMR website: http://mangia.caltech.edu/NMR.html

Retrieving Your Sample:

Following data acquisition, the instrument will automatically remove your sample and replace it with either the next queued sample or the standard. You may retrieve your sample from the black sample rack. If you do not see your sample there, please check the green sample rack adjacent to the robot. If it is not there either, contact a facility administrator or the GLA.

Switch Operator (Logout):

When not in use, VnmrJ should idle on the login screen. This can be done by selecting *File → Switch Operator* from the main page. ***Do NOT exit VnmrJ.***

Problems / Emergencies:

Please contact David VanderVelde (x3004) or the instrument's GLA if there are any problems with the NMR.

If your sample breaks in the probe, report the solvent and identity of the compound in your sample to lab staff so the probe can be promptly and safely cleaned. THIS QUALIFIES AS AN EMERGENCY.

If the D2O standard is broken or missing, please notify NMR personnel.

If the printouts have big black streaks on them, then the printer needs new toner. Notify NMR personnel.

Useful Commands

nt=###	set number of transients
bs=##	set block size
sw=####	change spectral width
d1=##	change relaxation delay
rts('standard') su	reload standard shim file
acqi	displays ACQI window, try this command if ACQI button does not appear.
su	type after rts command, after su acqproc sequence
wft	weighted fourier transform
aph	autophase
dscale / pscale	display/plot horizontal scale
nl	"nearest line" places cursor on nearest peak and indicates peak frequency
dres	shows digital resolution and peak width for a selected peak
f	"full," displays entire region of spectrum
text('xxxxxx')	add text to spectrum printout
axis='p' or 'h'	switches between ppm and hertz
vsadj	maximizes height of peaks in window
vs=##	"vertical scale," use to specify height maunally
vp=##	"vertical position;" set position of plot on page, must be ≥12 to plot integral values
sp=##p wp=##p	"Start plot" and "width of plot:" this command allows you to display a specified region. Ex: type sp=-.5p wp=12.5p to display the region between -.5 and 12 ppm.
ga or go	get acquisition, ga allows stoppage and resumption of acquisition.
sa or aa	stop acquisition (allows you to restart acquisition), abort all acquisition
ra	resume acquisition
cz	clear all integrals
svf	save file
jexp#	jump to another experiment window
bc	"baseline correct" – corrects baseline between integrated regions of spectrum
dpf / ppf	display/print plot frequencies
dpir / pir	display/plot integral reset values
pl	plots spectral line
pltext	plots text
pap	plot all parameters
ppa	plot minimal parameters
page	prints spectrum according to commands following pl
e	eject sample
i	insert sample
ds	display spectrum (interactive mode)

NMR Safety Guidelines

Compliance with these rules is required in the NMR Facility!

WARNING: Accidents caused by carelessness near strong magnetic fields can cause serious injury or death and significant damage to personal property, equipment and data

1. **Individuals with medical devices that can be disrupted by magnetic or radio fields (e.g. cardiac pacemakers and metal prostheses) must remain outside the 5-gauss perimeter.**
2. **In the event of a "magnet quench" (a sudden, violent release of cold helium gas from the magnet), evacuate the facility immediately, and contact NMR personnel.**
3. **Ferromagnetic metal objects must remain outside the 5-gauss perimeter, marked on the floor around each magnet.** This includes most ordinary tools, electronic equipment, compressed gas cylinders, steel chairs, and steel carts. Only non-ferromagnetic materials should be used near the instruments.
4. **Do not look directly down the upper barrel of an NMR spectrometer while trying to eject the sample.** Refer any problems with sample ejection to facility staff.
5. **Do not exceed the boiling or freezing points of your sample; approach these temperatures cautiously, as the instrument may overshoot the selected temperature or the temperature display may be somewhat inaccurate. This can cause excessive pressure to build up and break the tube.**
6. **If you have a sample that is expected to have or generate internal pressure, test all operations with it in a hood and with suitable safety equipment before inserting it into an instrument. Cold samples will warm up significantly while they are being inserted into the instrument, even if the probe is precooled.**
7. **Keep your hands out of the path of the robotic arm of the SMS sample changers.** Broken NMR tubes and cuts to your hands could result.
8. **Wipe down NMR tubes with a Kimwipe before they go in the magnet and <u>after</u> you remove any lab gloves you have worn from your own lab. Don't wear lab gloves while handling spinner turbines or typing on shared keyboards.**
9. **Don't pipet or mix chemical solutions on the computer work tables—use the fume hood.** Lab gloves and eye protection should be worn for those operations, but remove the gloves before returning to the spectrometer.
10. **Cards with magnetic strips, cellular phones, laptops and mechanical watches should remain outside the 5-gauss perimeter.** Strong magnetic fields surrounding the NMR spectrometers can damage the strip of magnetic media found on credit cards, ATM cards, driver's licenses, and other kinds of cards, disk drives, and other susceptible devices.

I have received a copy of these safety guidelines, read them, and will abide by them.

Name of Facility User: _____ Date: _____

IMSS Username: _____ PTA #: _____

Email Address: _____ Advisor's name: _____

Status: Postdoc____ Graduate Student____ Special Student/Visitor____ Other_____

Undergraduate, here this summer only: ____ or continuing Caltech student: ____

I have trained the above user in the proper use of the facility instruments and proper safety guidelines.

Name of GLA: _____ Instrument: _____ Date: _____ Account made: _____

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