

## General Information for Automated Inova 500 MHz NMR (Indy)

Primary Contacts:

NMR Facility Manager: David VanderVelde, x3004, [davidv@caltech.edu](mailto:davidv@caltech.edu)

GLA: Pamela Tadross, x6131, 646-220-5789 (cell), 204 Church, [ptadross@caltech.edu](mailto:ptadross@caltech.edu)

Additional contact:

NMR Facility Spectroscopist: Scott Ross, [scottr@caltech.edu](mailto:scottr@caltech.edu)

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The machine is currently capable of running  $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{19}\text{F}$ , and  $^{31}\text{P}$  samples. Do **NOT** run experiments on any other nuclei until you have been informed by the facility managers that the probe has been properly calibrated.

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### Inova 500 MHz NMR

Computer Terminal: Dell Precision, Intel Pentium 4

Operating System: Linux

NMR interface: VnmrJ 2.1B

### Sample Preparation:

All NMR tubes must be 8 inches in length, 5 mm in diameter. The autosampler can break tubes that are larger or smaller. Samples should contain 5 cm of solvent to ensure proper autolocking and autoshimming.

### Sign Up Rules:

Webcal sign-up for the automated 500 MHz NMR is located at:

<http://chemex.che.caltech.edu/cgi-bin/webcal-3/webcal.cgi?function=webday&cal=Indy>

and can be found by navigating from <http://chemex.che.caltech.edu/cgi-bin/webcal-3/webcal.cgi>.

The time limits are as follows:

Every day	8 am – 8 pm	1 hr
Every evening	8 pm – 12 am	2 hr
Every night	12 am – 8 am	8 hr

Please sign up for all experiments run on this machine, both daily and nightly, as you would when using Hg1 or Hg2.

## Start Up:

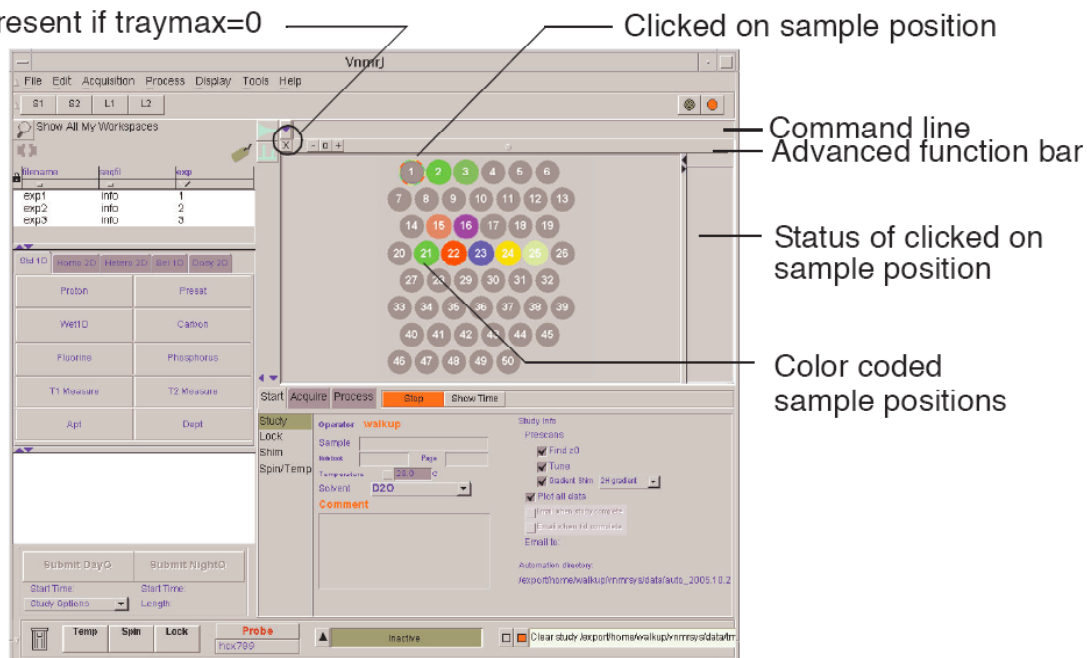
Click on the VnmrJ icon on the toolbar along the bottom of the desktop, or type *vnmrj* in the terminal window.

## Login:

On the login screen, select your research group from the *Operator* drop down menu. None of the research group operators require a password, so leave this space blank. Click *OK*.

## VnmrJ Interface:

Toggle is not present if traymax=0



## 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 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 test tube rack next to the autosampler tray, and use that spinner.

**Do NOT submit to slot #1. This is reserved for the standard.**

**If the autosampler malfunctions, it can be turned off by pressing the RED button.**

## Sample Submission:

Once your sample is in the rack, click on the corresponding slot number on the sample tray in VnmrJ. An animated circle should appear around that number.

Select your experiment from the list on the lefthand side of the screen. There are 4 tabs (Std 1D, Homo 2D, Hetero 2D, Sel 1D), each of which contains a list of experiments. Click on the experiment you want to run and it will be added to the StudyQ in the white space below. The title will read “new sample” until the sample has been submitted to the queue, at which point the title will become the location in which the FID will be autosaved.

Click all the experiments you want to run on the single sample you have selected. They will be listed underneath the title “new sample”.

In order to modify the acquisition parameters of an individual experiment, you must first double click on the experiment name (e.g. [1 min] Proton). If this is done correctly, the font for that experiment should change to bold and italics (e.g. [1 min] Proton → ***[1 min] Proton***). You may now make changes to the information under the *Start* and *Acquire* tabs directly to the right of the StudyQ.

Start tab:

- *Solvent*: select the deuterated solvent for your sample from the drop down menu
- *Sample*: text entered in the fourth line of the parameter array on the printout.
- *Comment*: same as *Text* command, add text to top left corner of spectrum

Acquire tab:

- *Spectral width (ppm)*
- *Relaxation delay (sec)*
- *Number of scans (nt)*

Click *Show Time* to see how your changes affect the acquisition time.

To remove an experiment from the StudyQ, click and drag it to the trashcan in the lower lefthand corner. Clicking on the blue experiment title (e.g. **Proton**) will remove that experiment for the chosen sample. Clicking on the “new sample” title will remove the entire list of experiments for that sample, effectively removing the sample itself from the queue.

When you are finished modifying the parameters of all the experiments for a given sample, you are ready to submit that sample to the DayQ or NightQ. Click *Submit DayQ* / *Submit NightQ*. The “new sample” title will change to the name of the directory to which your data will be saved and a padlock icon will appear next to each experiment listed under that title signifying that no further changes may be made.

**NOTE:** The StudyQ only shows experiments submitted under the current operator. To view a complete list of all experiments submitted in chronological order, type *status* in the command line.

The progress of the current acquisition may be monitored from the bar at the bottom center of the main page.

**All entries must be recorded in the logbook.**

### **Autosaved Data:**

As with Hg3, when each acquisition is completed the spectrum will be plotted automatically. In the parameters listed at the top lefthand corner of the plot, you will see three lines that look similar to this:

```
Automation directory: /home/autouser/vnmrsys/data/auto_2006.08.18_01
File: Proton01
Sample id: /home/operator/vnmrsys/data/Autosave_Files/2006Aug1801
```

The *automation directory* is where ALL data files are autosaved. However, it will be easier to find your data in your specific *operator autosave directory*, where only spectra acquired by your operator (e.g., research group) will be saved. This location is shown under the sample ID. So in the example above, the proton FID acquired on August 18, 2006 is saved under the file name “Proton01” in the directories listed on the first and third lines.

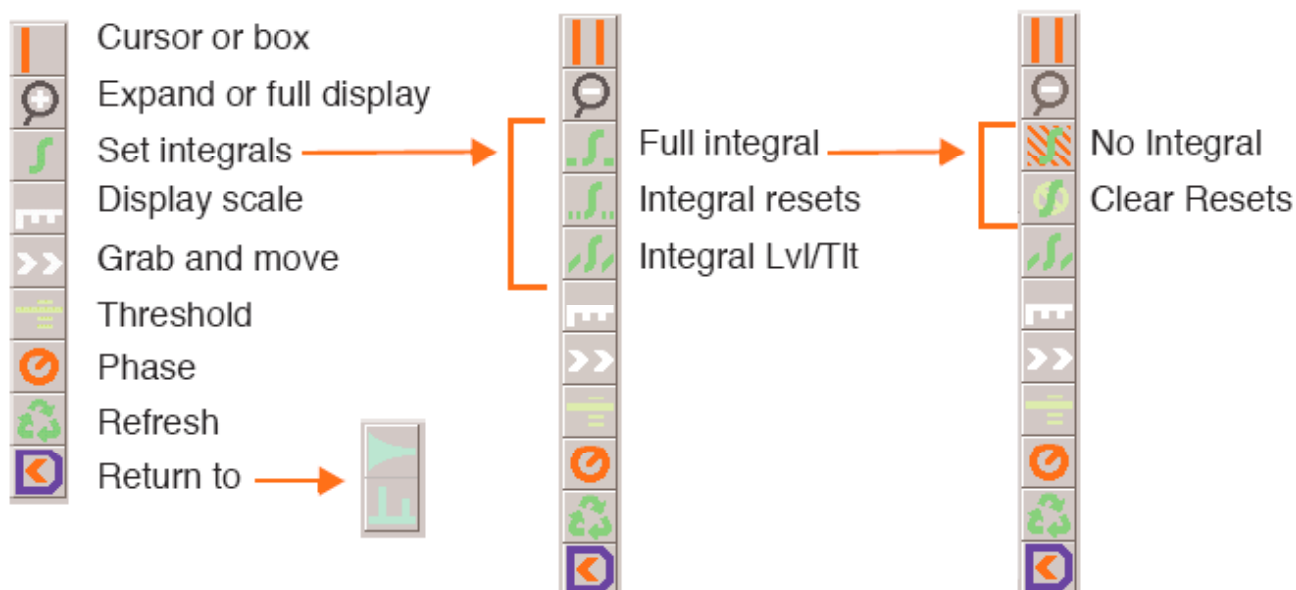
### **Data Processing:**

The easiest way to load your FID for processing is to click and drag it from the StudyQ. First click the X at the top lefthand corner of the sample tray, which should reveal a black screen (or possibly the pulse sequence of the most recently run experiment). Click and drag the line that has the experiment title and spectrum icon into the black window to load your spectrum. You may also need to use the *wft* command to bring your spectrum on screen. To get back to the sample tray, click the same top lefthand button (which will now be a O).

Under the *Process* tab (next to *Start* and *Acquire*), you may perform drift and baseline corrections, autofind integrals, adjust the display, etc.

All the commands that worked in Vnmr are also available in VnmrJ using the command line (e.g. *sp=##p wp=##p, vsadj, isadj, aph, dpf, dpir, dscale, setint(##),* etc.)

## Data Processing Toolbar:



The plot commands for VnmrJ are also the same as Vnmr. The following command will plot partial parameters, peak frequencies, integrals, and the scale for the portion of the spectrum currently on screen:

*pl ppa ppf pir pscale page*

## Saving Data:

You may save your processed FIDs as you would on Hg3. Directories have been made for each of the research group operators. Files should be saved in the directory */home/operator/vnmrsys/data*. An example of this would be:

```
/home/stoltz/vnmrsys/data/
```

You may make your own directory within the *data/* directory for your research group. To make the directory “gradstudent” within the “NMRfacility” group data directory, select File→Save and navigate to the *data/* directory. Then type in the full path name for the new directory:

```
/home/NMRfacility/vnmrsys/data/gradstudent
```

and press New Dir. You can also do this from the command line by typing:

```
mkdir(`/home/NMRfacility/vnmrsys/data/gradstudent/')
```

In order to save your processed data to this new directory, select File→Save and navigate to the directory. Enter a file name and press Save.

## **Retrieving Your Sample:**

If there are no experiments submitted after your own, the machine will idle with your sample in the magnet. If you would like to retrieve it, do so by replacing it with the D<sub>2</sub>O standard sample. This is accomplished by clicking the *Standard* button on the toolbar on the top of the main screen.

NOTE: You may receive the error message “seqfil ‘info’ cannot be found” when attempting to load the standard sample using the *Standard* button. If this happens, type “s2pul” in the command line and click the *Standard* button again.

## **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 Pamela Tadross (x6131 or 204 Church) if there are any problems with the NMR.