User's quick guide for Varian 500/400 NMR operation with VNMRJ 3.2 version

NMR is a very expensive instrument. Know what you are doing. Never do what you do not know how, don't even try it. Always ask if you are not sure.

Running NMR in vnmrj 3.2 is very friendly and easy. The operation has been configured in which it is very similar to the previous version with slightly different. Each regular user is a NMR operator under a group account walkup, a NMR operator does not have his/her own computer account, but the operator has to log into vnmrj with a password in order to run the NMR. There is no "New Automation" button available, but each operator has its own directory to store the data. Your data will be saved in the following directory structure:

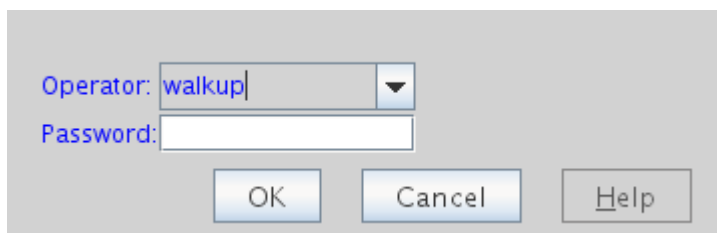/home/walkup/vnmrsys/data/username/samplename_date

Remember, it is your responsibility to back up your data. NMR lab is not responsible for any data loss at any situation.

Log in the computer with group username and passwd.

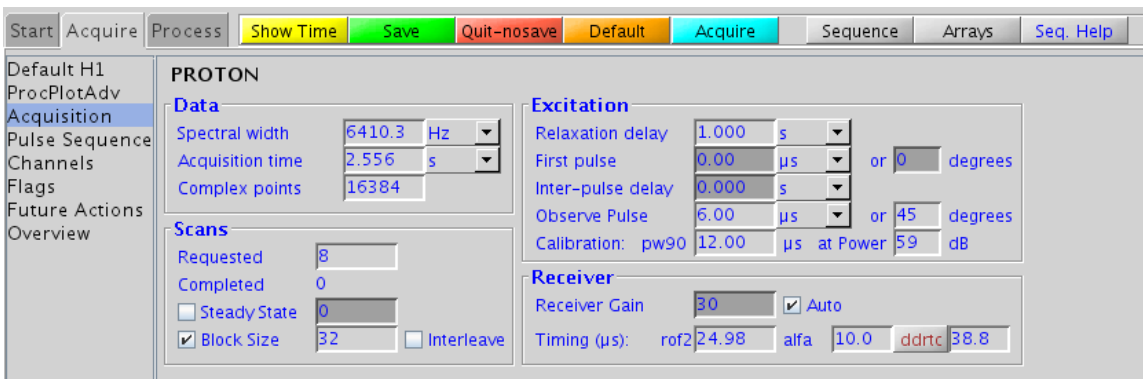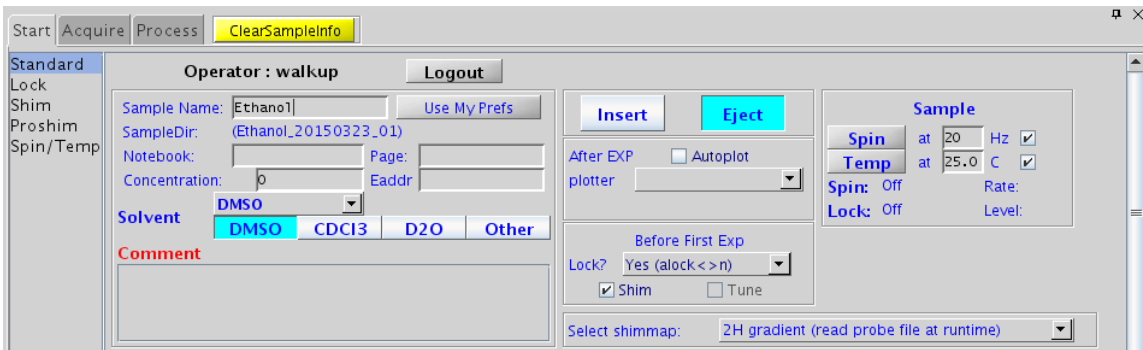Click vnmrj icon on the left side of the screen

You will see the vnmrj login window. select your login name at the drop down list, enter the passwd.and click "OK".

You will see the following window. Get familiar with this window with each button and its function.

menu bar
system toolbar
user toolbar
vertical panel

experiment
selector

list of
experiments

list of studies

Trash can

Command
line

Graphic
canvas

action bar

Parameter
panel

Message box

Acquisition status

1.   Click **New Study** tab.
2.   Click **Eject** button and remove the current sample.
3.   Place your sample in the spinner and adjust the position of the sample in the spinner.
4.   Place the new sample on the top of the upper barrel (top of magnet). Make sure that there is air flowing up. **Never place a sample on the top of magnet without air flow up, or serious damage will occur.**
5.   Click the **Insert** button.
Type in the sample's name, select a solvent and type in the comments. If you want to control temperature, check the Temperature and type in the temperature to be regulated (temperature control, your need additional training).

6.   Select an experimental protocol you want to run from the experiment selector panel (some most common 1D and 2D experiments are grouped in the "Common_NMR" tab. Say, you want to run 1D proton NMR, click **Proton,** the Proton protocol is loaded to Study Queue window. If you want to modify some of the acquisition parameter, such as the number of scans, spectral width, relaxation delay, you can double click the selected protocol in the study queue to load the protocol into canvas, pulse sequence is displayed. Click **Start** or **Acquire/Acquisition**, you can change any parameter you want.

Start | **Acquire** | **Process** | ClearSampleInfo

Standard
Lock
Shim
Proshim
Spin/Temp

Operator : walkup   Logout

Sample Name: Ethanol   Use My Prefs
SampleDir: (Ethanol_20150323_01)
Notebook: Page:
Concentration: 0   Eaddr
DMSO
Solvent
DMSO   CDCl3   D2O   Other
Comment

Insert   Eject

After EXP   ☐ Autoplot
plotter

Before First Exp
Lock? Yes (alock<>n)
☑ Shim   ☐ Tune

Select shimmap: 2H gradient (read probe file at runtime)

Sample
Spin   at 20   Hz ☑
Temp   at 25.0 C ☑
Spin: Off   Rate:
Lock: Off   Level:

Start | **Acquire** | **Process** | Show Time | Save | Quit-nosave | Default | Acquire | Sequence | Arrays | Seq. Help

Default H1
ProcPlotAdv
Acquisition
Pulse Sequence
Channels
Flags
Future Actions
Overview

PROTON

Data
Spectral width   6410.3   Hz
Acquisition time 2.556   s
Complex points   16384

Scans
Requested   8
Completed   0
☐ Steady State 0
☑ Block Size   32   ☐ Interleave

Excitation
Relaxation delay   1.000   s
First pulse   0.00   µs   or 0   degrees
Inter-pulse delay   0.000   s
Observe Pulse   6.00   µs   or 45 degrees
Calibration: pw90 12.00   µs   at Power 59 dB

Receiver
Receiver Gain   30   ☑ Auto
Timing (µs):   rof2 24.98   alfa 10.0   ddrtc 38.8

If you want to run 2D HMQC experiment, click g**Hmqc**. The gHmqc is loaded into the study queue. Make sure all the parameters are modified according to your sample.

Select as many protocols as you want and make adjustment on any parameters.

Always select a gradient version of the 2D protocol if it is available. In the example above, you should select the gHmqc protocol.

You may delete the protocol by drag it to the trash can.

Study Queue
Submit Queue   Done
Ethanol (to be submitted)
SampleInfo [12:20]
Proton_001 [0:28]
Carbon_001 [9:52]

New Study
Submit   to   Foreground
Clear Pending Exp from Queue

Temp   Spin   Lock   Sample

Click the protocol and drag it to trash can
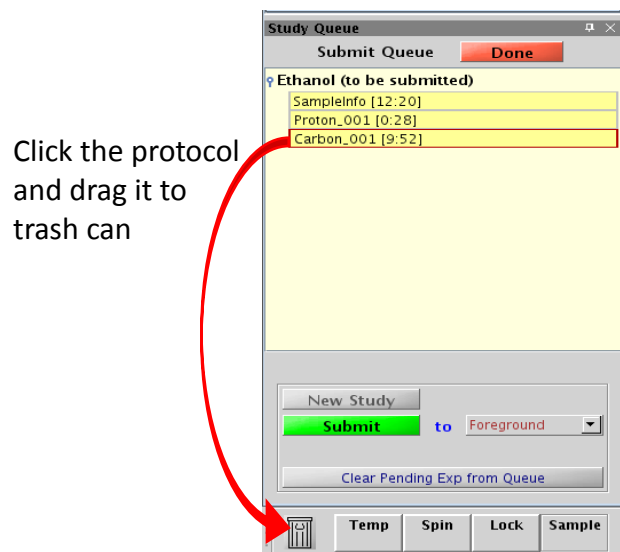
8.   After finishing protocol selection and parameter adjustment, click "**Submit**" to start to run all those experiments. At this point, all protocols are locked, no change can be made. But you a stop the run by clicking the red stop button.
9.   When all experiments are completed, all data are automatically processed and saved. Your data is in the directory

/home/walkup/vnmrsys/data/username/samplename_date

The data filenames will be **Proton.fid** and **Hmqc.fid**.
10.   Take out the sample and put a standard sample into the Magnet. Close the vnmrj program (you logout the vnmrj).
11.   You may reprocess your data at the workstation when it is available or transfer the data to your flash drive and use third party software to process it.

All spectra have to be plotted manually, no auto plot in this operation.

Arrayed NMR experiments

Arrayed experiments are very common such as in studying kinetics, you want to acquire NMR data at certain time intervals.

Let's say you want to acquire proton NMR data every hour for total of 5 hours with 6 NMR experiments.

Setup proton experiment with the above procedure.
At command line, type
pad=0,3600,3600,3600,3600,3600
pad is a parameter for pre acquisition delay. Click **Submit** to acquire a set of NMR data. The first value of pad is 0, which means that it will immediately acquire first data, then, wait for 3600 seconds (1 hour) to acquire second data, etc.

You may array other parameters, but you have to know what you are doing.
After the experiment is completed, type **ds(1)** to display the first spectrum (or any other spectrum), you may process this spectrum, (all other spectra will be automatically processed according to this spectrum).

Under the "Process" tab, there are many functions can be used (demonstrated below). Click "Display" to manipulate the spectrum and click the "Plot" to manipulate the plotting.
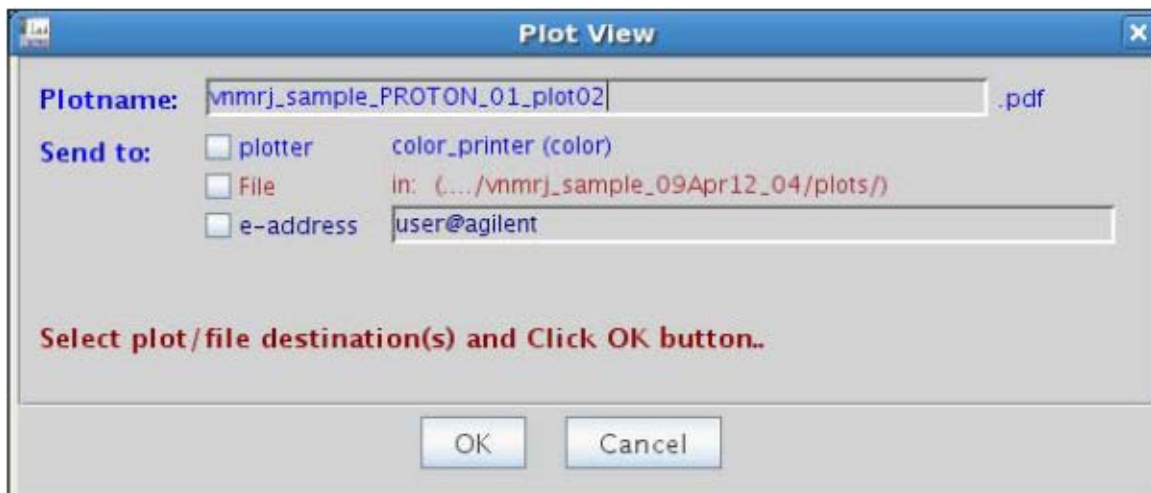
Display:
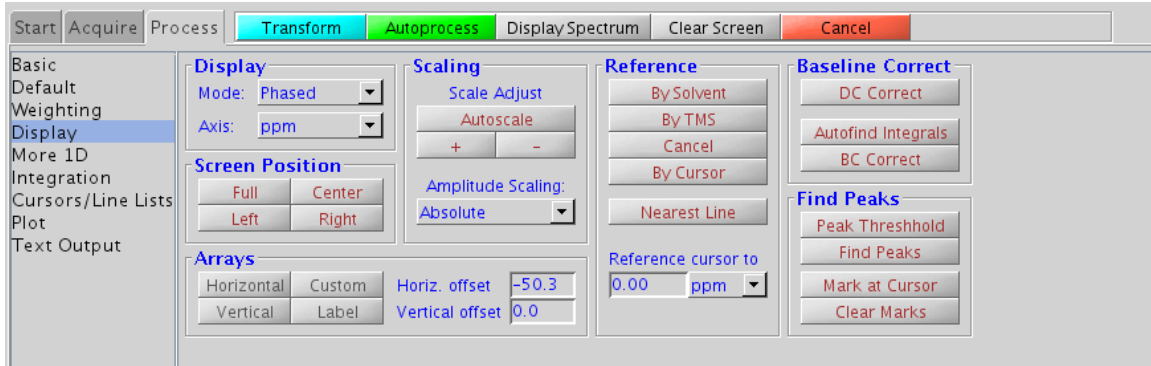
Plot:
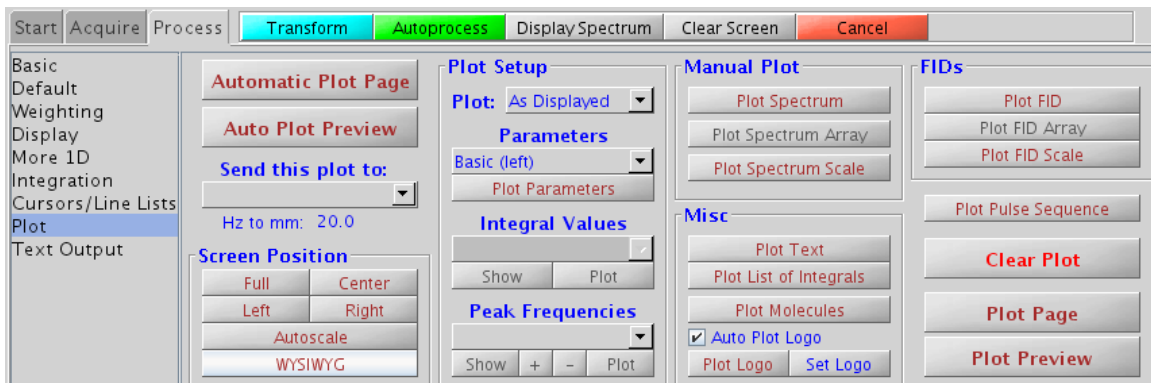
Save spectrum in PDF format

In the Plot panel, click those plot buttons you want to plot such as "Plot Spectrum", "Plot Spectrum scale" ……., click "Plot Preview", the Plot View dialog box appears,

check "File", then click "OK", the PDF plot spectrum is saved in the directory "Plot" under that sample name directory.

Create Inset in your plot and saved in PDF file:
1. Process the spectrum and get it ready for plot (such as phase, reference and integrals, etc..)
2. Click "Plot" at the left panel to open the plot panel, click all plots you want (such as "plot spectrum", "plot scale", …….).
3. Click "Curser/Line Lists" at the left panel, put two red cursers on the region of the spectrum you want to inset, click "Inset spectrum", the selected region will move up, click ⬚ on the top manual bar, move the mouse on the inset, click/hold/move the left mouse button, move the mouse, the inset will move with the mouse, move the inset to the positon you want, right click/hold/move the mouse, the inset will expand, you adjust the height of the inset by hold/move the middle mouse. The vertical position is controlled by parameter "vp", type vp? To see the current position value, assume it is 80, , type vp=100 will move the inset up to "100". When you are happy with the inset, click "Plot inset".
   If you want to make second inset, click " Return from inset", put two red cursers on the region, click "inset spectrum", the selected region will move up, click ⬚ on the top manual bar, move the mouse on the inset, click/hold/move the left mouse button, the inset will move with the mouse, move the inset to the positon you want, right click/hold/move the mouse, the inset will expand, When you are happy with the inset, click "Plot inset". Repeat for more insets.
4. Click "Plot" at the left panel, click "Plot Preview", the Plot View dialog box appears, save the file with the name you entered.
If the plot is not you want, click "Clear Plot" and start over.

Example of two insets
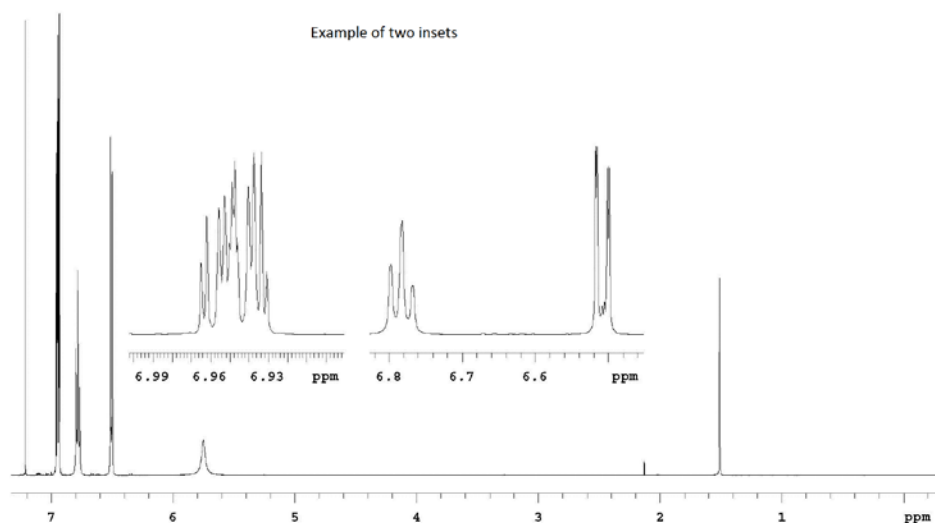
Use external reference

In many cases that there are no internal reference peak in the NMR spectrum, so you need to use an external reference. Here is the instruction.

1. Setup and run your NMR sample as usual (such as make a new study, or lock and shim…..)

2. When it is finishes, save your spectrum (if run New Study, the data is automatically saved), take out your sample, insert the reference sample. (do not make new study, this way you use exact the same parameters as your sample)

    > At the command line, type **alock='n' wshim='n'**, hit enter (so it will not do auto lock and auto shim, keep the condition exact the same as your sample)

    > If the concentration of the reference sample is high, you expect that the signal will be strong, you do not need run so many scans, type **nt=4** (8, or 16 whatever you think is appropriate)

    > Type **ga** to get the NMR spectrum of the reference sample.

3. After the reference sample is finished, phase the spectrum, locate the reference peak (the shape of the reference peak may not be so good, but it is not important), check if its chemical shift value is correct or not. Let's say that the correct chemical shift value of the reference is 4.50ppm based on previous papers or experiments, but in this experiment, the chemical shift value of the reference peak is 5.1ppm, so this is not correct, this tells you that in the spectrum of your sample at the position of 5.1ppm, it should be 4.5 ppm, so go to the spectrum of your sample, locate the position 5.1 ppm and change it to 4.5 ppm.

4. Take out the reference sample and start your next sample with normal procedures (such as make a new study, etc) . If it needs external reference again, follow the steps above.

Variable temperature NMR

In the normal set up condition, the cooling and heating air uses house compressed air which passes through the air dryer with dew point of -40 ºC. Therefore the lowest temperature that the preconditioning unit FTS system can be set is -40 ºC without ice accumulated in the pre conditioning unit. The lowest temperature can be achieved at the NMR sample is -30 ºC. It is necessary to use N2 gas to do lower temperature NMR experiment. For high temperature NMR, it is limited to 40 ºC if using house air without damaging the probe. For temperature higher than 40 ºC NMR, it is necessary to use N2 gas to heat the NMR sample.

Click **Spin/Temp** button, you will see the following window.
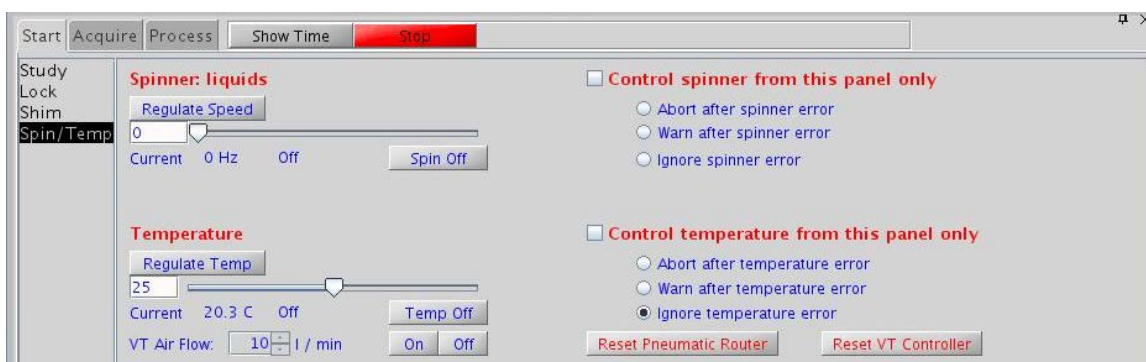
Check the "Control temperature from this panel only and you may check one of the choices below that. Click "Reset VT" Click Reset Pneumatic Fault. Make sure there is air flow at the pneumatic box. Check the flow meter "Gas flow sensor"
Type the temperature you want to be regulated below the **Regulate Temp** button, press return after you input the value. Click the **Regulate Temp** button. Go to FTS preconditioning system near the magnet.
Turn on the power of the system, press up or down arrow button to set the temperature 10 degree below the regulated temperature, then press **Run** button. If the temperature is not regulated, you will see "NotReg" below the temperature control slider. If the temp is regulated, you will see "Reg". You can see the actual sample temperature next to it.

Wait for at least 5 minutes to let the temperature equilibrium through the sample after the temperature is regulated before acquiring NMR data.
To change to the next temperature, type in new value and press return key, and click **Regulate Temp** button. Go to FTS control unit, change the temperature setting.

For temperature below -30 ºC or higher than 40 ºC, additional N2 gas set up is needed, which is not available now.