

# CDD 600 MHz S User Manual

Version 1.2

May 2005

Notes on Console: New Varian NMR System console (S for short) has Direct Drive architecture that eliminates quadrature detection. The digitization in this console reduces the noise in 2-D spectra. The need for spinning is also greatly reduced with this console.

**IMPORTANT:** To do phosphorous or fluorine experiments, the probe will require re-tuning by trained personnel. Check the status before attempting any of these experiments. Please read all rules and policies before operation.

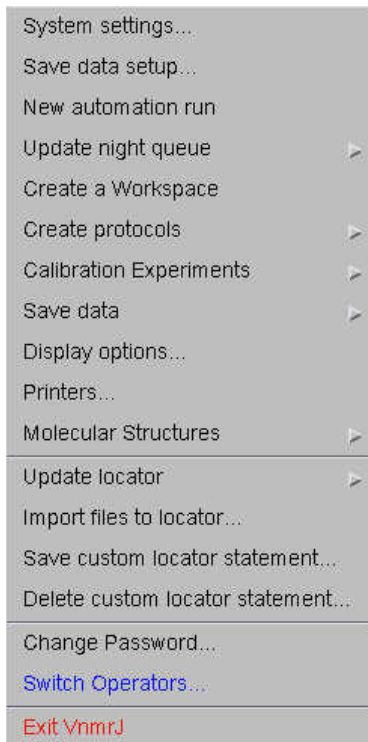
## Getting started as a walkup owner account:

1. Login to the workstation.
2. On the CDE bar, select VNMRj icon to start the software. (Click only once!)

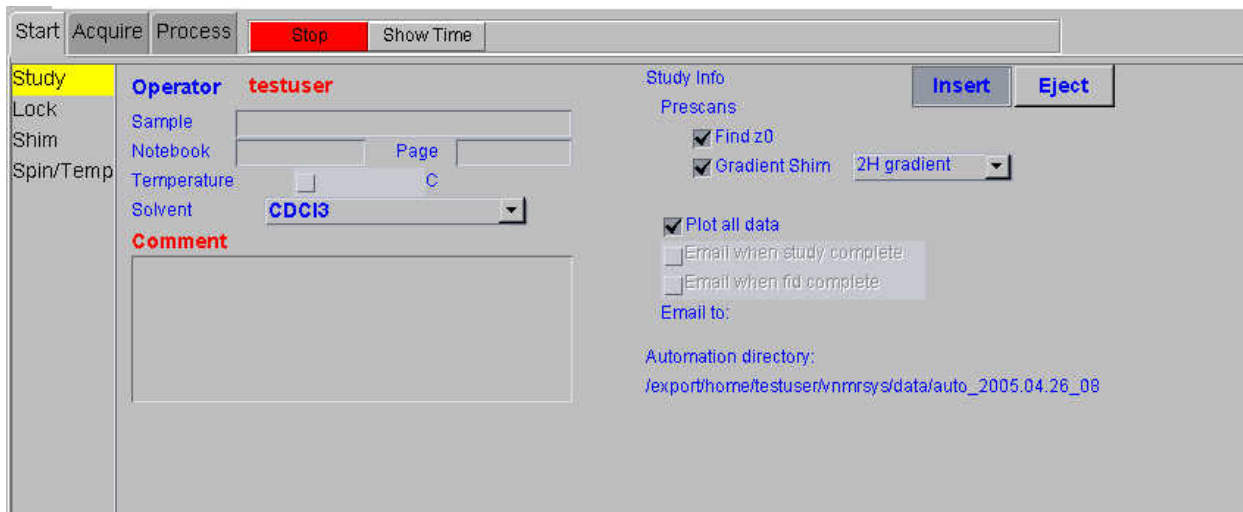


3. Opens in the directory (/export/home/username).
4. From menu, select **Utilities** → **New automation run** (This should be done once a day to avoid generating extra directories. If it is performed more than once a day, an extra automation directory is generated called auto\_date\_01 or \_02 etc. depending on how often it's done.)

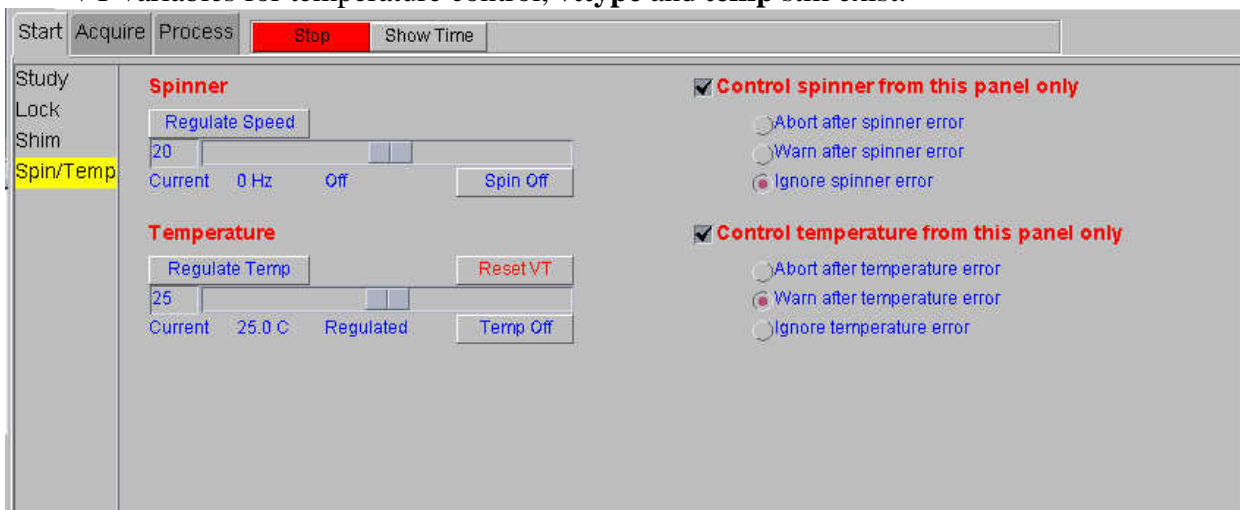
## Utilities Menu:



- Fill out Start (Study) menu for sample name and text. Be sure to select the correct solvent. Use this window to **[Eject]** the current sample and **[Insert]** the new sample. Be sure to **always** use the depth gauge to adjust the sample height in the spinner before putting the sample in the magnet. Select **Find z0** and **Gradient shim [2H gradient]** for autolock and autoshim. If autoplots are not desired, uncheck the “Plot all data”.



- Use the Spin/Temp menu to change the temperature or turn the spinner off or on. To change the temperature, drag the bar or type in an exact value in the window. Choose **[Regulate Temp]** to start the temperature change. Wait for the temperature to regulate before continuing. Please re-set the temperature to 25 deg. C at the end of experiments. VT variables for temperature control, **vttype** and **temp** still exist.



- Drag experiments into study queue. Total run time is indicated in the lower window.

## Study Queue:

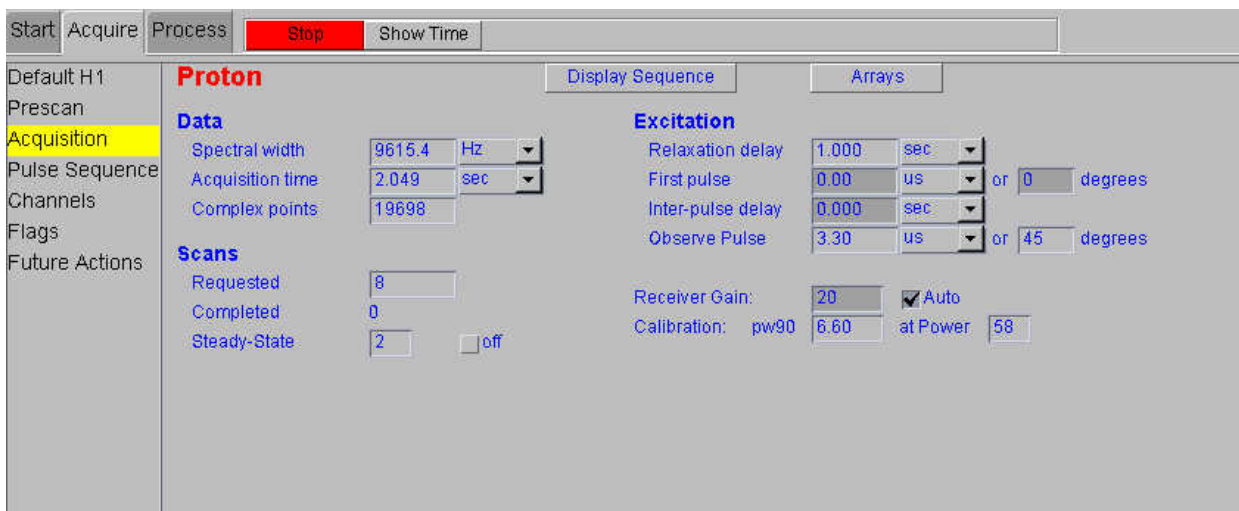
The screenshot shows the 'Study Queue' window. At the top, there are tabs for 'Std 1D', 'Homo 2D', 'Hetero 2D', and 'Sel 1D'. Below these are several rows of buttons: 'Proton' and 'Presat', 'Wet1D' and 'Carbon', 'Fluorine' and 'Phosphorus', 'T1 Measure' and 'T2 Measure', and 'Apt' and 'Dept'. The main area shows a tree view for 'new sample' with 'Proton' and 'Carbon' sub-items. 'Proton' has a '[1 min] Proton' sub-item, and 'Carbon' has a '[10 min] Carbon' sub-item, which is highlighted in yellow. At the bottom, there is a 'Submit' button and a 'Study Options' dropdown menu showing 'Length: 14 min'.

8. Double click the time portion under each element (i.e. [1min]Proton) to edit the parameters of the experiment in the Acquire menu on the right. Wait a moment for the computer to load the parameters before continuing to the Acquire tab.

The screenshot shows the 'Acquire' window for the 'Proton' experiment. The 'Default H1' tab is selected. The 'Proton' experiment name is displayed in red. The 'Display Sequence' button is visible. The parameters are as follows:

Parameter	Value
Spectral Width [ppm]	14 -> -2
Downfield	14.0
Upfield	-2.0
Pulse Width [degrees]	45
Enter pulse angle	45
Relaxation Delay [sec]	1
Number of Scans	8
Spin	<input checked="" type="checkbox"/> 20 Hz
Transform size	32k
Line Broadening [Hz]	0.2
Plot when done	Spectrum: Full
Parameters	Basic, Top Left
Peak Values	
Integrals	Partial

9. In the **Acquire [Default H1]** menu, select the spectral width in ppm, set the number of scans, turn the spinner off, set the relaxation delay, and turn off plotter or integrals if desired.
10. In the **Acquire [Acquisition]** window, the acquisition time, steady state scans and receiver gain may be set if desired.



11. Use the **Acquire [Future Actions]** menu to check the save path for the file(s).
12. In the command line, use `>su` Tune the proton channel and carbon channel, if required.
  - \*\* Do not attempt this if you are not sure how!!!\*\*** Use channel 1 on the tune box for proton, adjust the proton tune and match (same wand, says “Proton”) to at least 100 or less (about 50 is the minimum). Tune channel 2 for carbon, tune and match are on separate wands (labeled X-tune and X-match) to at least 100 or less. Be sure that attenuation is at nine. Never force the wands! If they seem tight or won’t move, stop and ask for help. A proton experiment defaults to channel 1 proton and channel 2 carbon for the S console so proton and carbon can be tuned from a 1-D proton experiment set-up.
13. To start the experiment, select the **[Submit]** button under the study queue. The experiment(s) will now run. The deuterium ( $2H$ ) gradient shimming will occur first followed by the autolock before the actual experiments begin. While the experiments are running, the progress can be monitored in the acquisition status window accessed at the bottom of the screen.

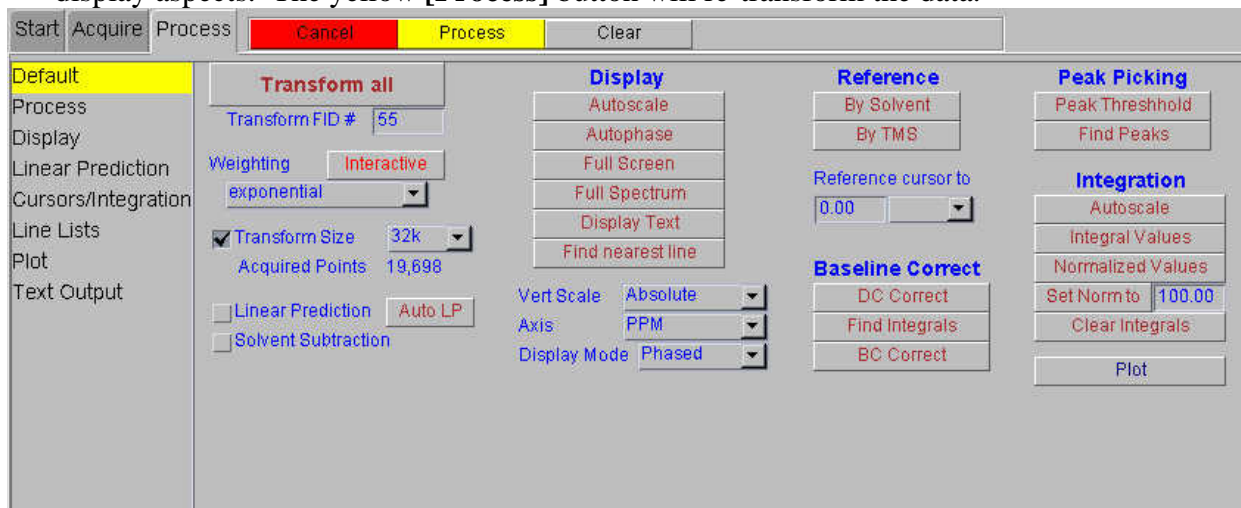


In the study queue, an experiment is highlighted in blue when it is running and in yellow when waiting in the queue.

14. To start another experiment, use **[Study Options]** → Clear study page and new sample. Then begin again. (Do not use Utilities → new automation run.) Instead fill in a new sample name and drag experiments into the queue.
15. When finished collecting data, replace the standard sample and lock it by typing `>autolock` in the window. Wait until the autolock finishes (it should beep). Then in the menu (top) select **Utilities** → **Exit VNMRj**. Exit the account.

## Processing

- The last experiment run will be left processed on the screen at the end of the run. To process a specific experiment, double click on the time title in the study queue for a specific experiment. Otherwise, load it from the file manager by dragging it into the display window.
- To change processing parameters, use the Process tab. Under the Process tab in the **[Default]** menu, spectra can be referenced and peak thresholds set as well as some display aspects. The yellow **[Process]** button will re-transform the data.




- The side bar menus are sometimes the quickest, easiest means for processing. The interactive menu (shown) will show an explanation of each button if the cursor is placed over it.




## Referencing:

Put the cursor on the resonance to be referenced, typically solvent peak. In the **Process->Default** menu select **[Find nearest line]** or use **>nl** in the command line to put the cursor on the top of the peak. In the **Process->Default** menu pull down the menu under “Reference cursor to” to select ‘ppm’ and enter the reference value in the window, hit return.

## Reset Integrals

In the side bar menu, chose Part Integral . The green line(s) for integration should appear. To clear the reset points, use >cz in the command or choose [clear integrals] in the

**Process->Default** menu. Then use select 'resets' from the side bar menu . Use the left mouse button to add reset points and the right mouse button to remove resets. The spectrum can be expanded to more exactly set the reset points.

After adding all the reset points, place the red cursor over the integral region to use for normalization. In the **Process->Default** menu enter the value for that integral in the box for "Set Norm to" and hit return. Then click on [Set Norm to]. Click on [Integral Values] to display the integrals on the screen.

## Plot data

All the plot commands still work from the command line so those are one option for plotting. To plot similar spectra with menu buttons, use the **Process->Plot** menu. Select each item to be included in the plot and always end with clicking on [Plot Page] to send the plot to the printer. The word "RAST" will appear up in the display screen near the sequence info when plotting information is in the buffer. An example for plotting: [Plot spectrum] [Plot scale] select parameter printout, [Plot text], select peak frequencies on peaks, [Plot Integrals], for integral values [Plot Scaled], [Plot Page]. Any combination may be used.

## To load data:

Data will be automatically saved in the *username/vnmrsys/data* directory in a directory called *auto\_date*. Under the *auto\_date* directory will be a directory with the sample name. In that directory are the actual data files that end in .fid.

Data can be loaded by dragging the .fid file into the display space or double-clicking it.

## To change your password:

In VNMRj, select **Utilities** → **Change Password**

## To change the display colors:

In VNMRj, select **Utilities** → **Display Options**

## Experiments on Study Queue Menus:

1-D options:

Proton	Presat
Wet1D	Carbon
Fluorine*	Phosphorous*
T1 Measure	T2 Measure
Apt	Dept

Homo 2-D experiments:

Cosy	Dqcosy
------	--------

Noesy	Roesy
Tocsy	Gcosy
Gdqcosy	

Hetero 2-D experiments:

Hmqc	Hmbc
Hsqc	Ghmbc
Ghmqc	Ghsqc
Hmqctoxy	Hsqctoxy
Ghsqctoxy	Cigar2j3j

Sel 1-D

Noesy 1D	Roesy 1D
Tocsy 1D	

\*These experiments require re-tuning of the probe. Please ask for assistance.

## Quick Sheets

### Proton 1-D

1. Login, open VNMRj.
2. Utilities→New Automation Run (first run of the day)
3. Start - > Study Menu
4. Insert new sample.
5. Select solvent, enter sample name, text, check plotting or not, check temperature. Be sure Find Z0 and Gradient Shim [2H gradient] are checked.
6. Pull Proton experiment into study queue. Double click exp. Time for Acquire parameters.
7. Acquire→Default H1 menu: change # of scans, relaxation delay, spectral width.
8. Tune proton (Channel 1) on the probe.
9. Check study length and click Submit. Wait for exp. to finish. Amount of time remaining shows in acq status window (blue bottom).
10. Spectrum will autoprocess and appear in the window. File will be autosaved in filename found under Acquire→Future actions menu.
11. Use side menu to adjust integrals, threshold, scale, expansion etc.
12. Replace sample with dummy D2O sample and run autolock.
13. Log out of VNMRj Utilities→Exit VNMRj. Log out of account.

### Carbon 1-D

1. Start - > Study Menu. Select solvent, enter sample name, text, check plotting or not, temperature. Use Find Z0 and Gradient Shim [2H gradient] only if new sample.
2. Pull Carbon experiment into study queue. Double click the time for Acquire parameters.
3. Use Acquire→Default C13 menu: change spectral width (ppm), # of scans, relaxation delay, decoupling mode (default Decoupled +NOE).
4. Tune the carbon channel (or X-channel), Channel 2.
5. Check study length and click Submit. Wait for exp. to finish. Amount of time remaining shows in acq status window (blue bottom).
6. Spectrum will autoprocess and appear in the window. File will be autosaved in filename found under Acquire→Future actions menu.
7. Use side menu to adjust integrals, threshold, scale, expansion etc.
8. Replace sample with dummy D2O sample and run autolock.
9. Log out of VNMRj Utilities→Exit VNMRj. Log out of account.