# Introduction to VnmrJ

This brief introduction to VnmrJ can be used for running the Varian 500 and 700 MHz machines in our facility. Procedures particular to a machine are described as such in the test. This manual is meant to be just a quick guide for running these machines. User are encouraged to use the ***VnmrJ Liquids*** and ***VnmrJ Command and Parameter Reference*** manuals for more detailed description of anything mentioned in this introduction.

# *Introduction to the VnmrJ Graphical User Interface*

Once logged on to Linux, you can start VnmrJ in one of two ways:

a. From the terminal window: type **vnmrj** at a terminal window and press enter.
b. From the CDE toolbar: click on VnmrJ icon in the CDE toolbar.

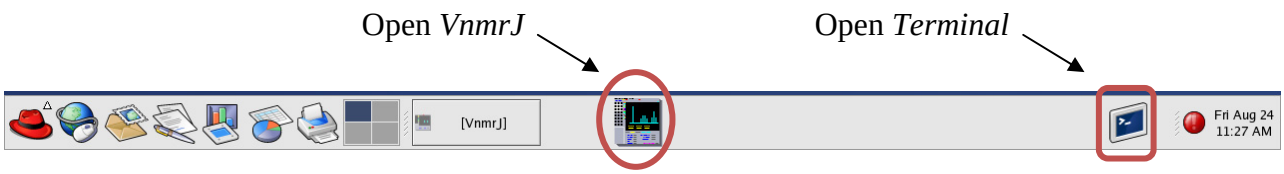



**Figure 1 Starting *VnmrJ* from CDE toolbar.**

The *VnmrJ* experimental liquids interface you will see when you start the software is shown below (Figure 2). The tabs on the right side of the picture (under the graphics canvas) are the ones you will be using most of the time. The functions of some of VnmrJ buttons are described below.

## VnmrJ Buttons and their Functions

**Menu Bar:**
Use this bar in operations related to data acquisition, processing, display, and plotting. The menu bar also provides access to little-used features, settings, and preferences.

**Advanced Function Bar (command line):**
This is where you type VnmrJ commands. The commands you enter would most of the time be for things that can't be accessed from the Graphical User Interface.

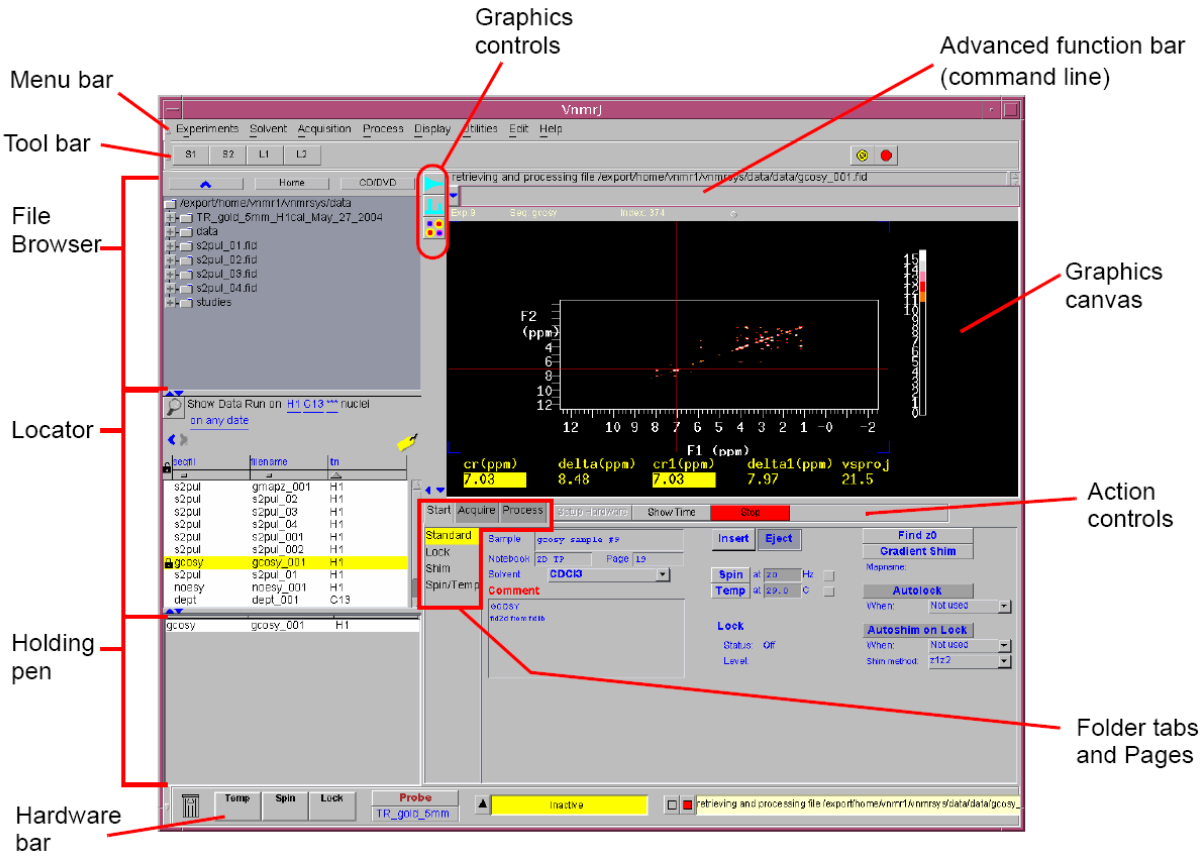



**Figure 2 *VnmrJ* Experimental Liquids Interface**

**Tool Bar:**

The tool bar is directly below the menu bar. These buttons provide quick access to common functions. The following tools are the default available in this tool bar:


Save the current locator data sort display. To save the display, click the button for three seconds. To return to the saved display, click again on the button.

.

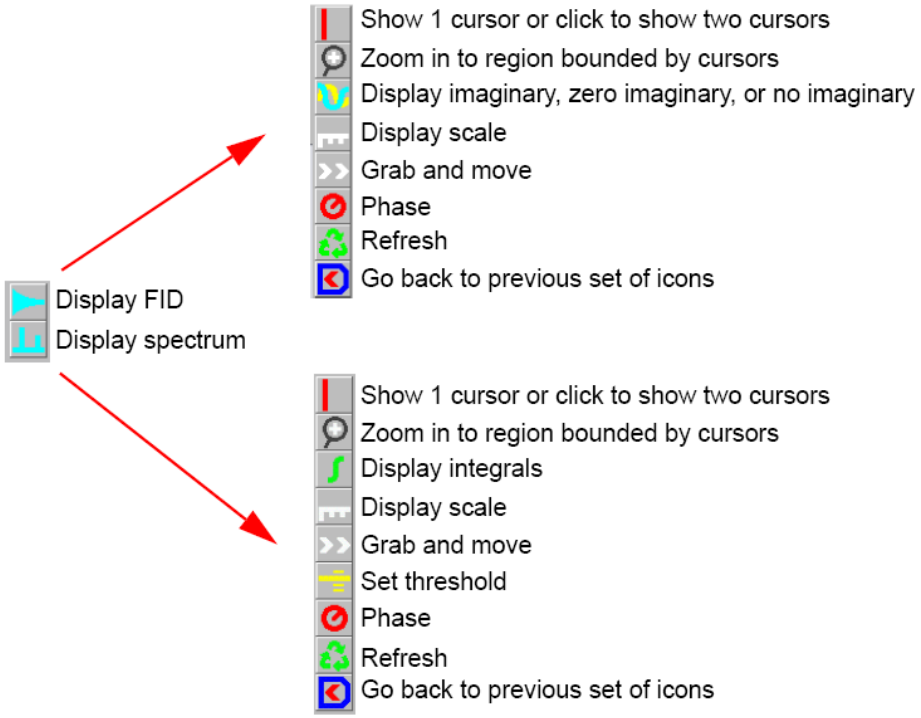

Save the current screen layout (graphics, a parameter panel, locator sizes). To save the layout, click the button for three seconds. To return to the saved layout, click again on the button.

 cancels commands.

 stops acquisition.

**Graphics Canvas:**
This is the place where your fids/spectra and pulse sequences are displayed.

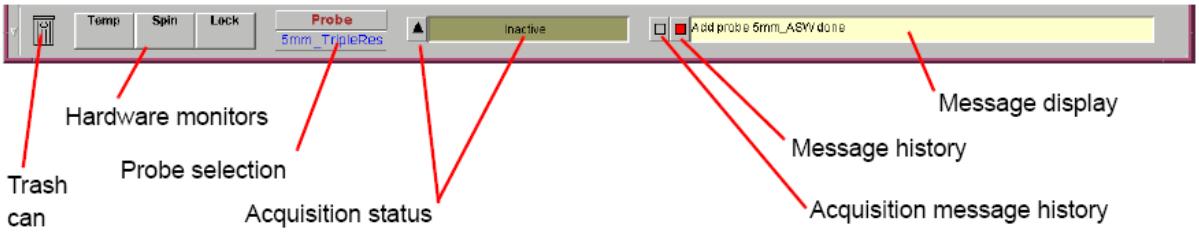
**Graphics Control:**
These buttons control many commands used to view and manipulate fid/spectrum displayed on the graphics canvas.




**Folder tabs and Pages**:
These are the most used and most important buttons. Most of the commands you need to setup, acquire, and process data are included in these buttons.

**Hardware Bar:**
This is where acquisition status is displayed. If there is no running experiment, the acquisition status should indicate ***'Idle'***. The ***message display*** window is where you would see messages about the current experiment or responses to you VnmrJ command

line queries. For example, if you type ***time*** on the command line, you will see the time it takes to finish the current experiment at the ***message display*** window.




## 1.1 NMR Experiment Tasks

### Set Up an Experiment

Set up the experiment using the pages in the **Setup** tab.

1. Select the **Setup-Lock** page.

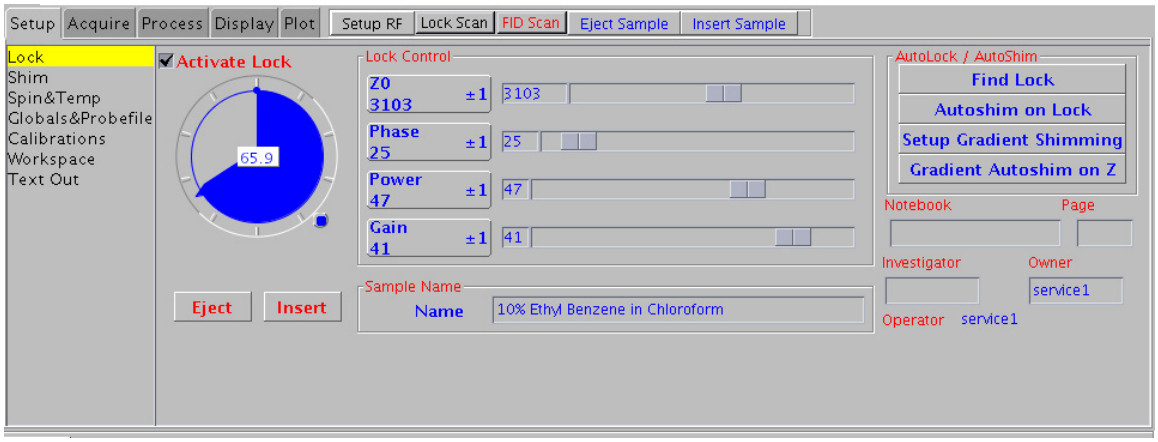



**Figure 3: Setup GUI**

   If you want to name your sample, enter a name for the in the **Sample** field. You can further define your sample by filling in the **Notebook** and **Page.**
2. Insert the sample.
3. Regulate spinning and temperature on the **Spin/Temp** page.
4. **Find Lock** adjust the lock using the **Shim** and **Lock** pages.
5. Shim the system to adjust the field homogeneity using the controls provided on the **Shim** page.

### Acquire a Spectrum

Set acquisition and acquire data using the pages in the **Acquire** tab.

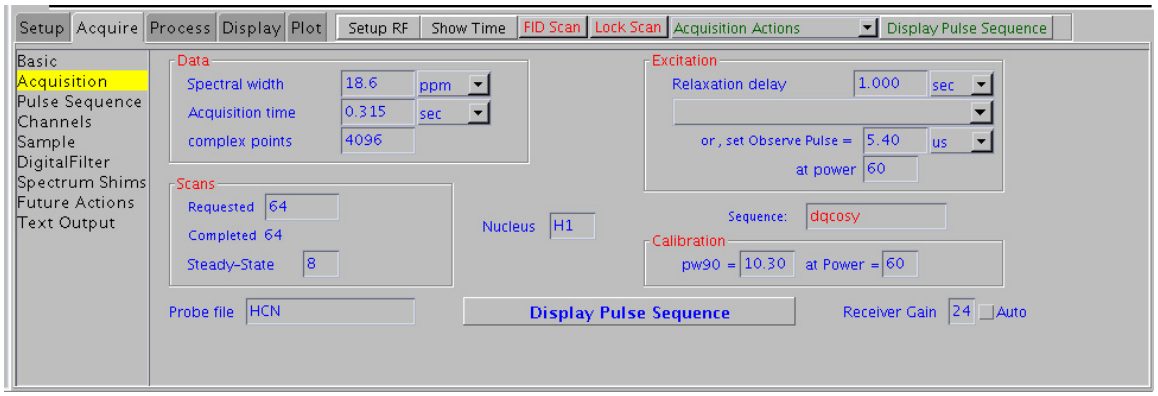



Figure 4

1. Set up experimental parameters and post acquisition actions. To enter solvent name click **Sample** and fill the solvent box.
2. Click the **Acquisition Action** button and select ***Acquire then process*** to acquire NMR data.

## Process the Data

Process the NMR data using the pages in the **Process** tab.

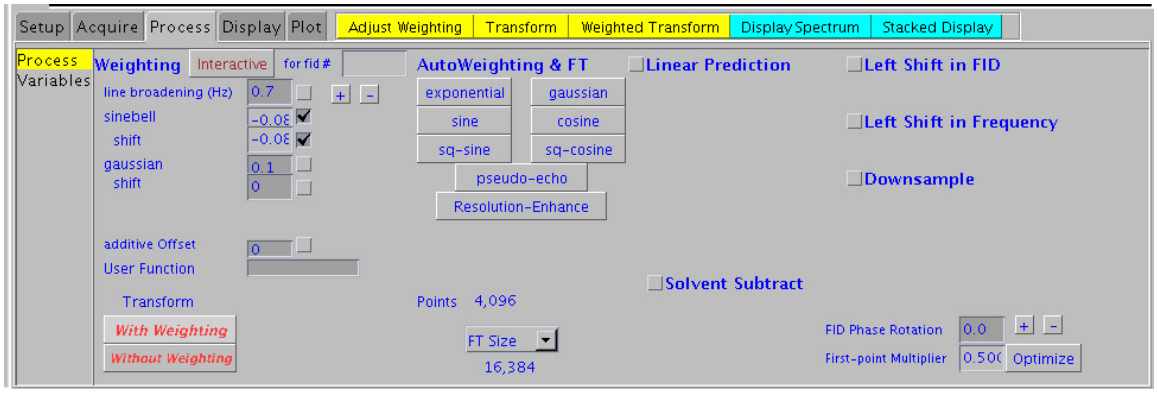



**Figure 5**

## Display the Data

Use the **Display** page and the graphic control buttons to manipulate the display of the NMR data.

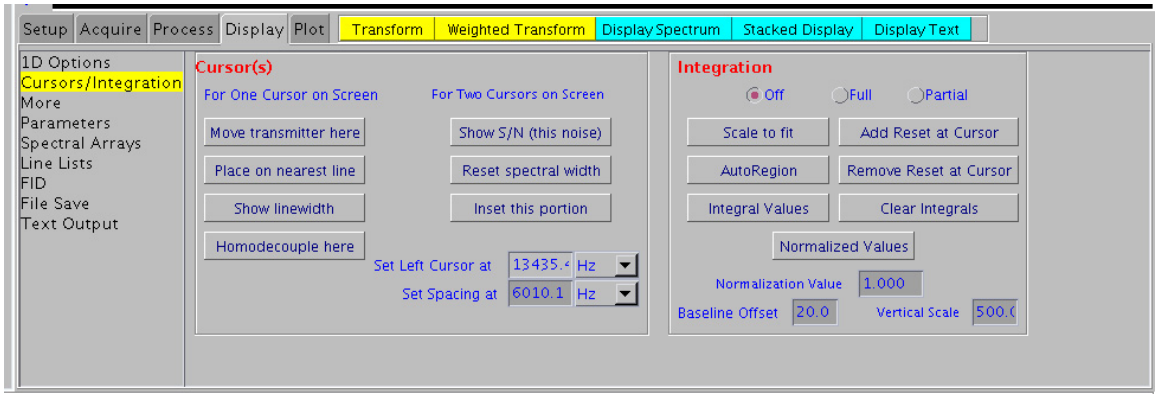



**Figure 6**

## Print or Plot the Data

Use the **Plot** page to create a print or plot. You can also add sample information such as concentration, solvent, temperature etc on the *Sample Information* box.

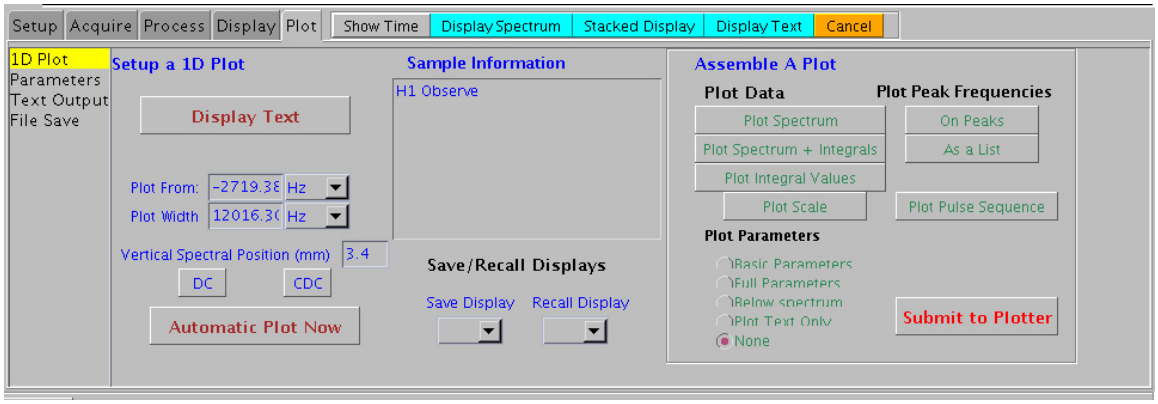



**Figure 7**

## 1.2 Saving NMR Data

If you acquired the data but did not select the **Automatic FID save** feature in the **Future Actions** page under the **Acquire** tab and you now want to save the data, you can save the data using either the Save Data Setup window or the Future Actions page.

| *Method* | *Description* |
|---|---|
| Future Actions Page | Click on the **Acquire** tab, **Future Actions** tab, and **Save FID Now** button. If you checked the **Automatic FID Save** in the **Future Actions** panel before starting data acquisition, the data has already been saved when acquisition completes. |
| Menu<br>Utilities -> Save data | Click **Utilities** -> **Save data** and select either:<br>Save data setup -- to customize where and under what name data is saved<br>Save current fid -- to save the FID.<br>Save process as -- to enter a name under which to save the process. |

## 1.3 Stopping an Experiment

There are four ways to stop an experiment:

• Clicking on the **Stop** button

.

• Clicking on **Acquisition Action (Figure 10)** then select **Abort Acquisition** from the drop down menu.

• Enter **aa** on the command line

## *2. General Acquisition Setup: 1D Proton*

Entering sample information

Enter a name that will be used for the saved dataset. Additional fields (notebook,page) are optional.

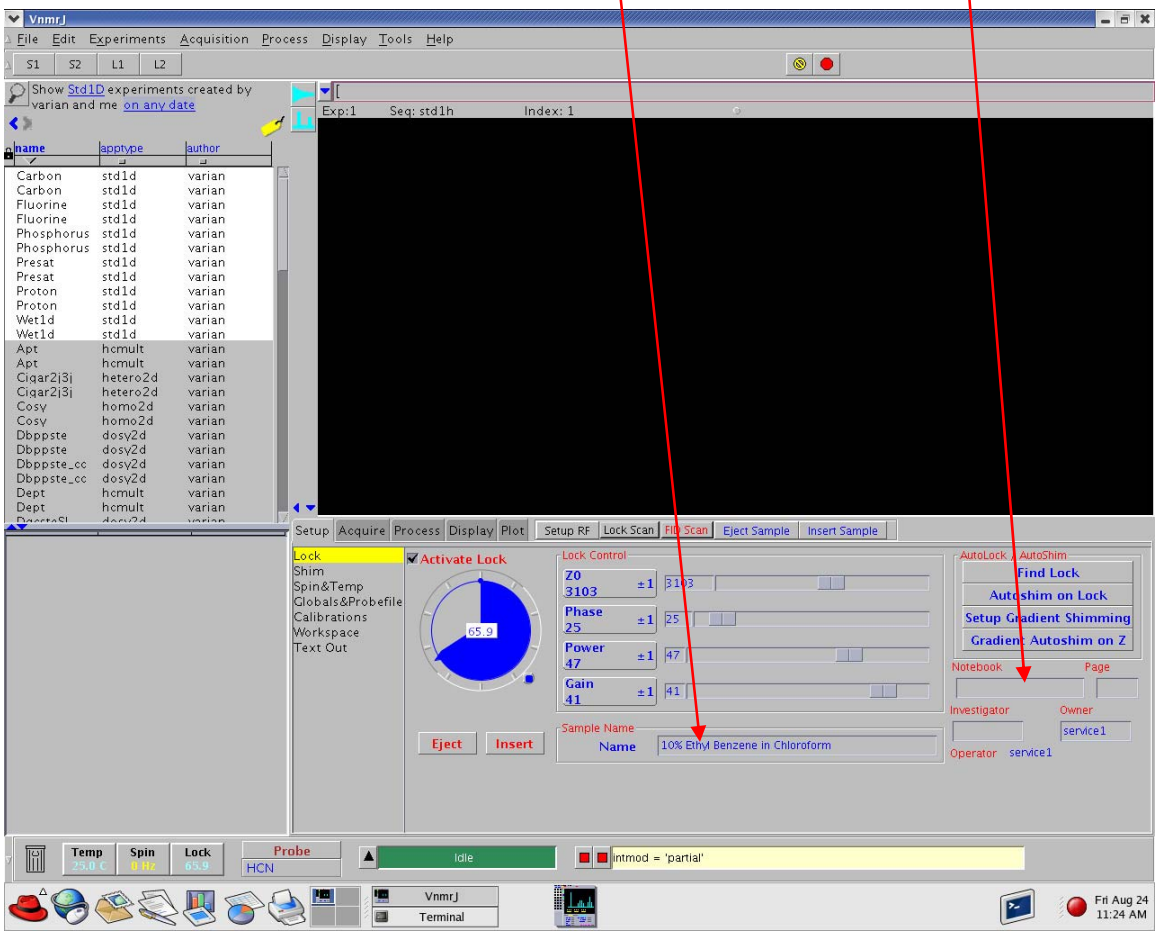



**Figure 8**

A text box is available under **Plot-> 1D Plot** for entering a detailed description of the sample. This text will be saved with the data.

### 2.1 Preparing the Sample

Prepare your sample in a deuterated solvent to a final volume of ~ 0.6 ml for use in 5 mm NMR tube. The most common deuterated solvents are D2O, deuterated acetone, chloroform, methylene chloride, and DMSO. Solid materials in your sample cause field inhomogeneity and degrade the quality of your NMR spectra. Filter out any insoluble materials from your sample before you transfer it to your NMR tube.

***Sample Position Using the Depth Gauge:***
Insert the turbine into the top of the sample depth Gauge and insert your NMR sample in to the turbine. Gently push your sample tube down until it touches the movable bottom of the sample depth Gauge. Center the sample volume in the dotted box (receiver coil area) of the Gauge.

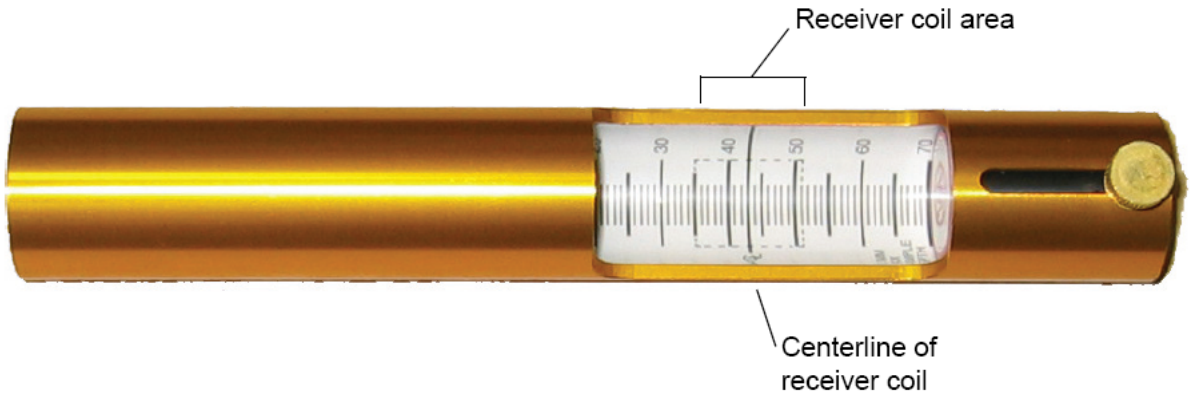



**Figure 9 Sample Depth Gauge**

Inserting Sample:

To insert your sample into the magnet, first click on **Eject Sample**, wait for a loud sound of a rushing air and then put your sample into the top of upper barrel. of the magnet after you hear. Then Click on **Insert Sample** to let the sample descend into the probe.

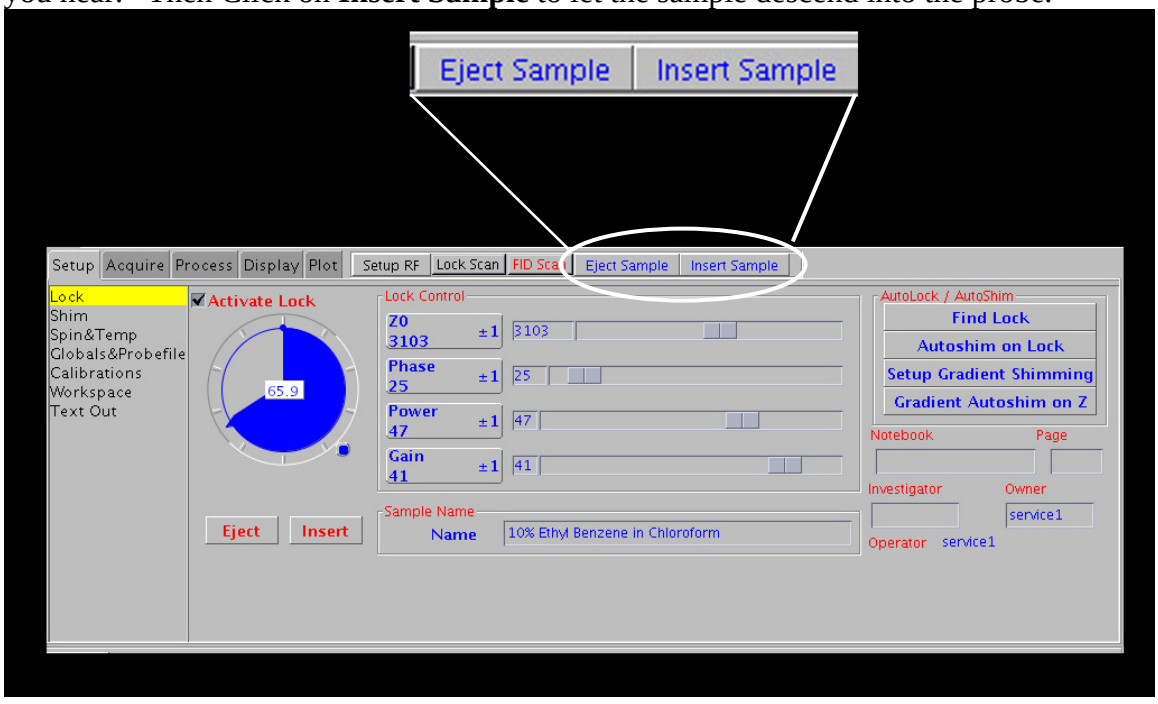



**Figure 10**

## 2.4 Temperature and sample spinning.

To setup the temperature go to, **Setup-> Spin & Temp** sub-panel**,** enter the desired temperature and click **Regulate Temperature at this Value**.

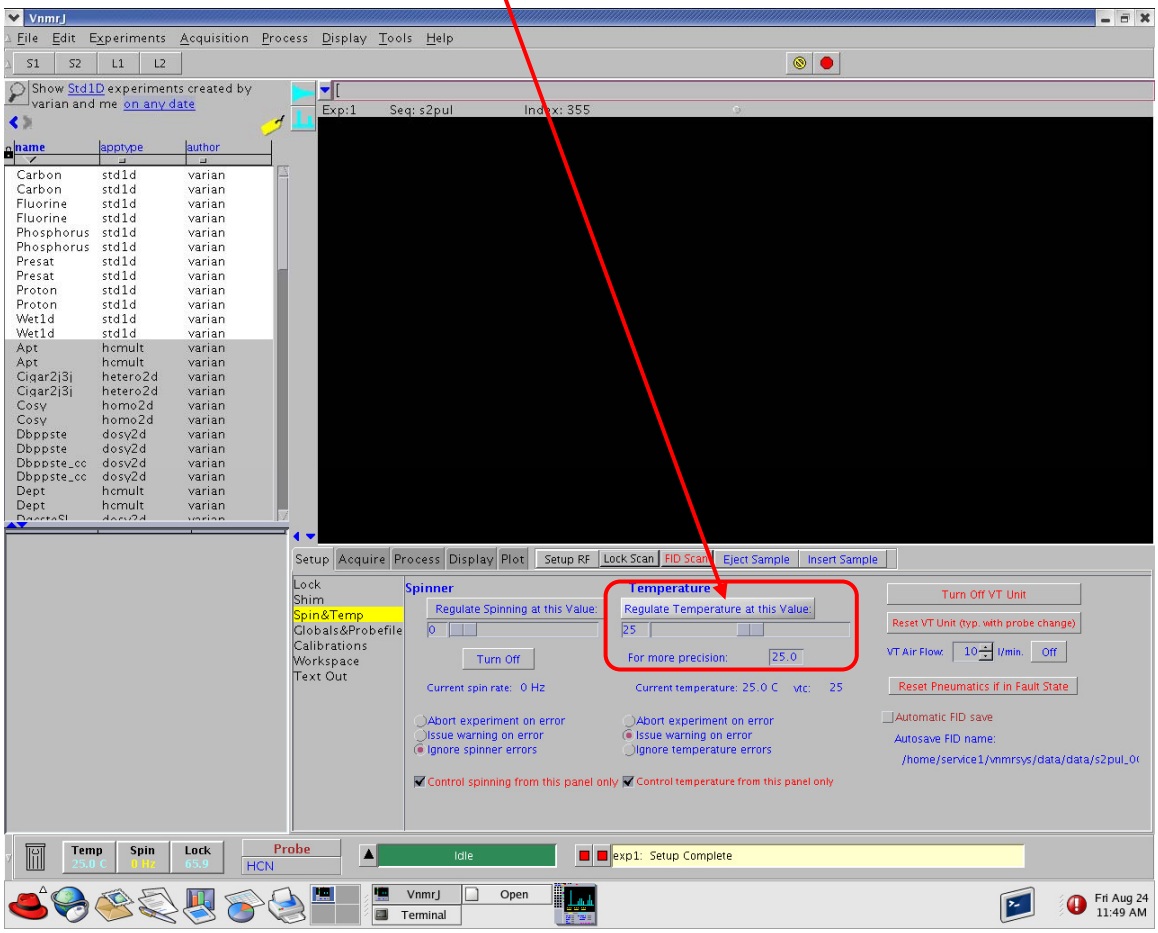



**Figure 11**

The default temperature is 25 $^{0}$C and we don't spin samples (set Spin Rate to zero).

## 2.2 Probe Tuning

Tunning the 700 MHz Probe:

To tune the HCN probe on the 700 MHz magnet, type **mtune** on the VnmrJ command line. Then select **Setup->Probe tune** (Figure 12) and select the Channel you want to tune ($^{1}$H is Channel 1, $^{13}$C is channel 2 and $^{15}$N is channel 3) then click **Start Probe Tune** (Figure 12)**.** The Tune GUI will changes to what is shown in Figure 13. Click on **Autoscale** (Figure 13) and you will see the tune signal which is the same thing as the wobble signal you are familiar with from your experience with Bruker machines (Figure 14).

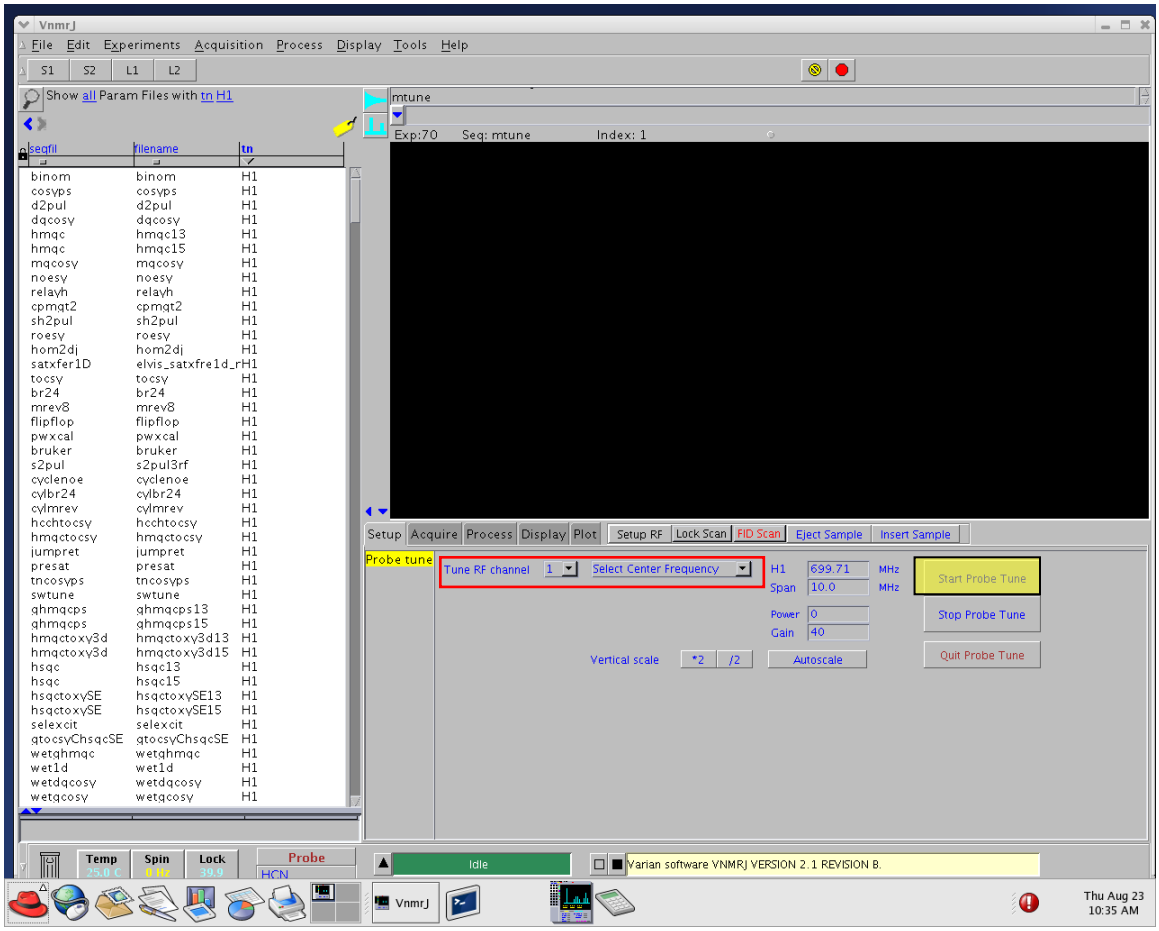



Figure 12: Probe Tune Interface you will see after typing **mtune** at VnmrJ command line.

The $^{1}$H channel has both tune and match modules. The $^{13}$C and $^{15}$N and $^{2}$H channels, however, have only tune module.
To tune the $^{1}$H channel, pull down the **tune rod (Figure 15)**, make **1H-T** active by rotating the plastic part above the **tune rod**, push the tune rod up and rotate it clockwise or counterclockwise so the center of the tune signal displayed on your monitor (Figre 14) coincides with the green line at the center. Once the tune signal is centered around the green line, pull down the **tune rod** and rotate the plastic rod to activate **1H-M** to do impedance matching. Rotate the **tune rod** such that the bottom of the tune signal is as close to the x-axis as possible. If the tunning is affected by matching (tune signal not centered around the green line any more) repeat the tunning and matching process one more time.

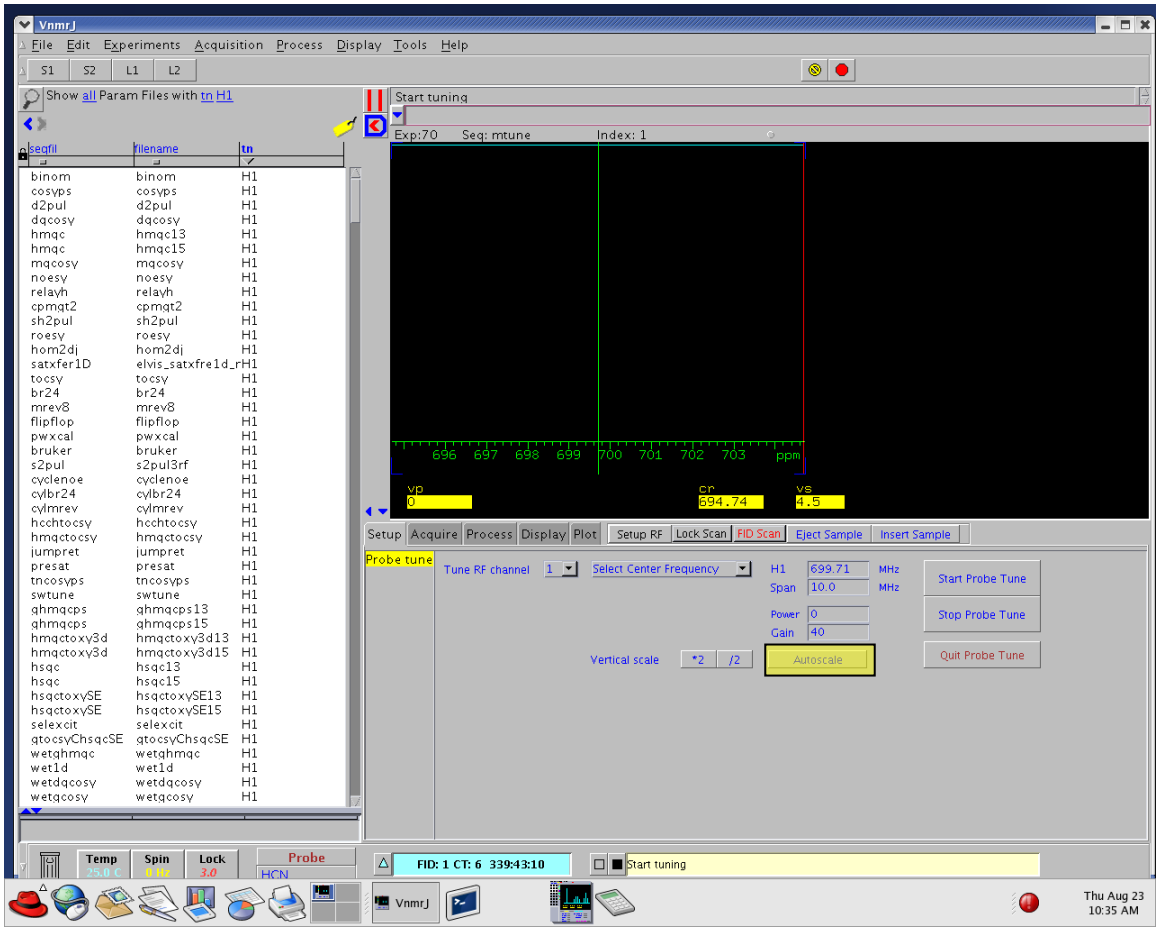



Figure 13: Tune GUI after initiating tunning by clicking on **Start Probe Tune.**

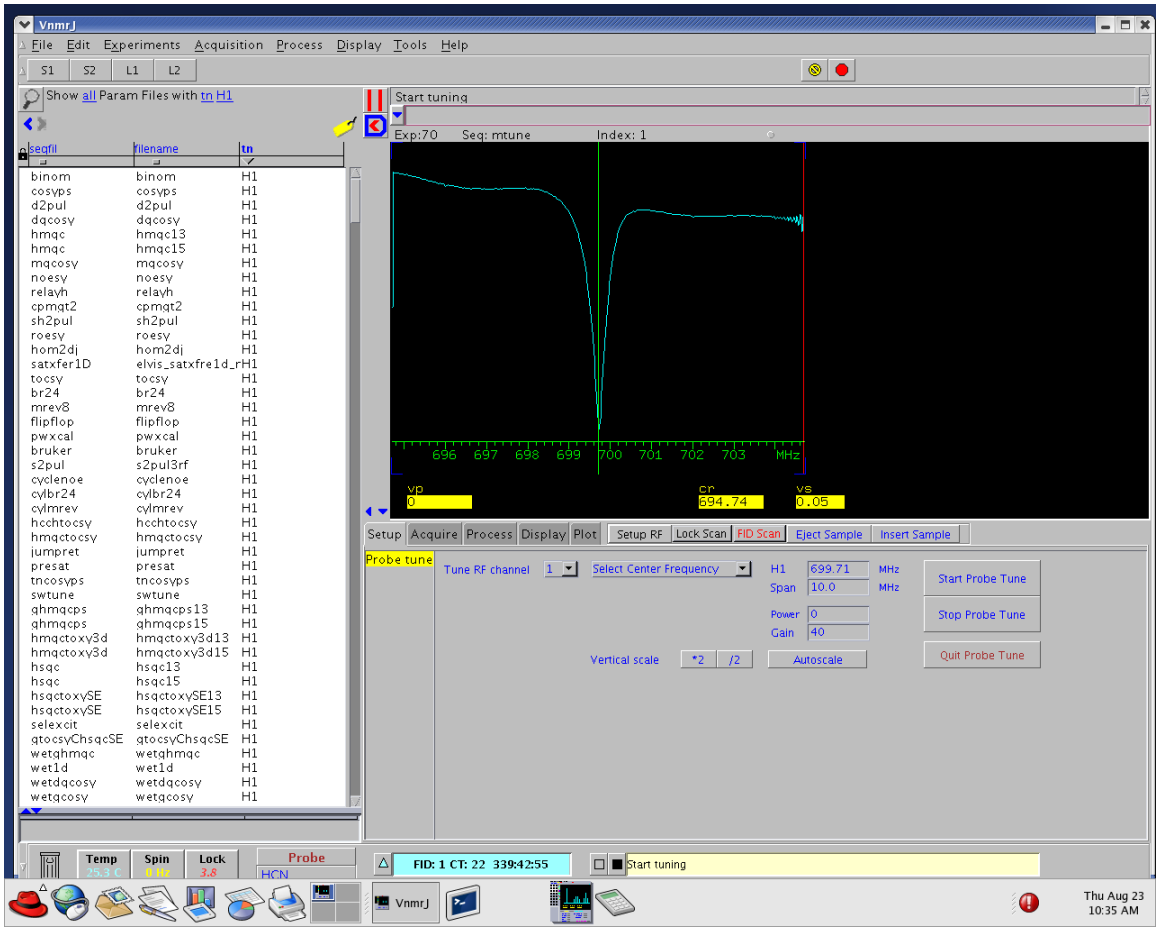



**Figure 14: Tune GUI: Autoscaled. When the probe is tuned and matched, the tune signal is collinear with the vertical green line and its tip is as close to the baseline (horizontal axis) as possible.**

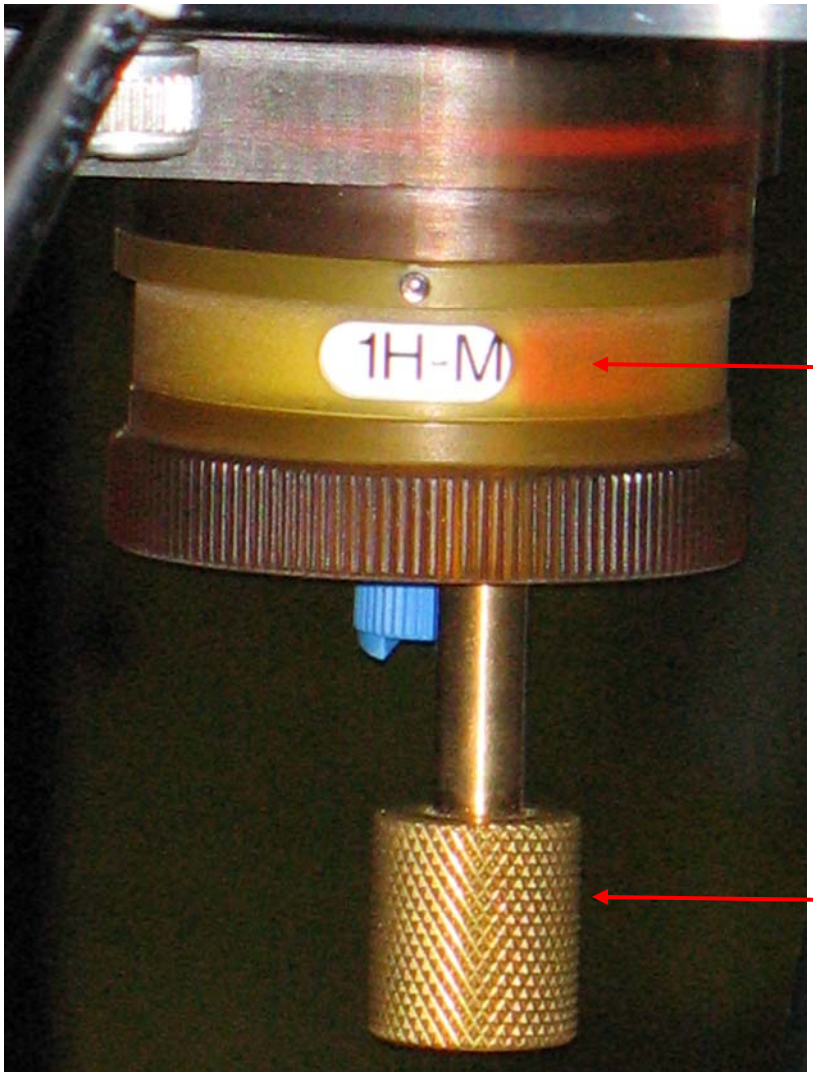



**Figure 15: The tunning/matching rod for the HCN cold probe is located at the bottom of the magnet.**

## 2.2 Locking

Go to **Setup->Lock** and click on **Find Lock** to set the lock frequency for the solvent chosen. Click **Lock Scan** to see lock trace. If it has sinusoidal character, adjust Z0 to make it flat (Figure 16); otherwise click **Activate Lock**.

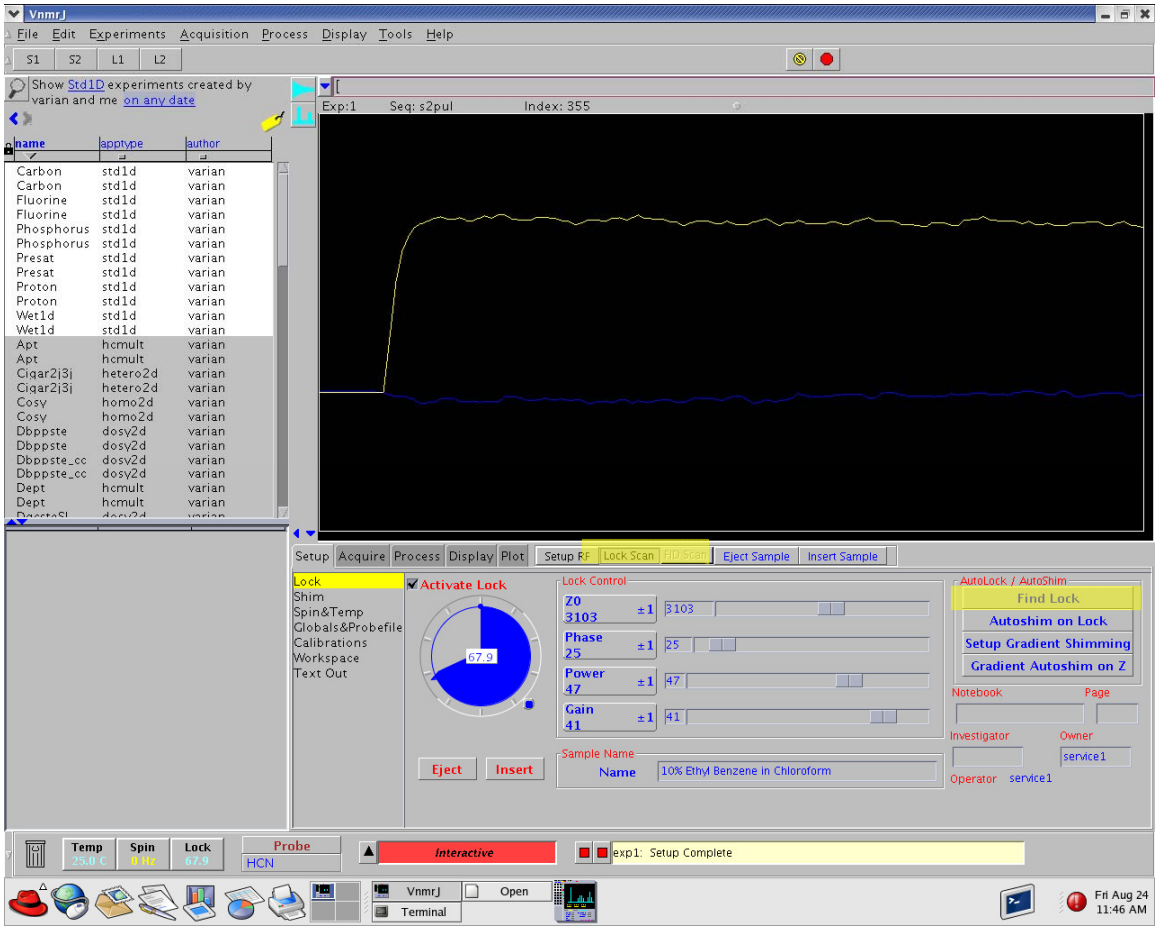



**Figure 17: When the lock frequency is right, the lock signal is a flat line with a step.**

Click **Lock Scan** again to remove lock display

## 2.3 Manual Shimming

Click on **Setup->Shim** subpanel. Reduce lock **power** and **gain** (keep them roughly equal) so display shows lock level between 80 and 95.

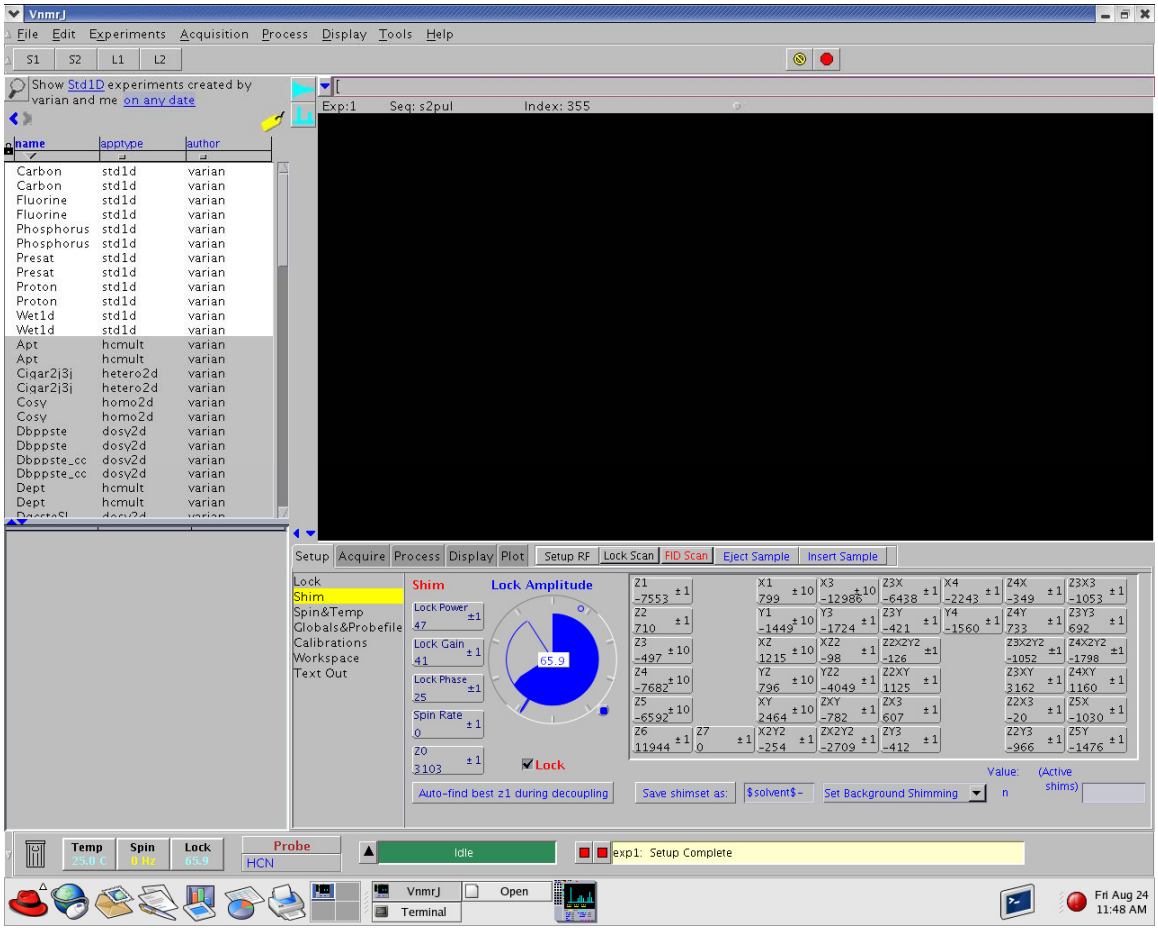



**Figure 17: Shim panel**

Adjust Z1 (either right button + or left button -) to increase lock level.
The middle button switches between 1,10,100X. If the level goes above 100, reduce lock gain. Continue until you can no longer increase the lock level. Switch to Z2 and do the same. Return to Z1, and repeat. Stop when no increase in lock level is observed with either Z1 or Z2. Switch to Z3 and maximize lock level. Go back to Z1 and Z2. Optimize X, Y,XZ, YZ and XY and go back to Z1, Z2 and Z3 one more time.

## *3. Proton 1D: Standard parameters*

Join experiment-1 by typing ‘jexp1’ at the VnmrJ command line. To open 1D Proton protocol, Double click **Proton** in the locator protocol list.

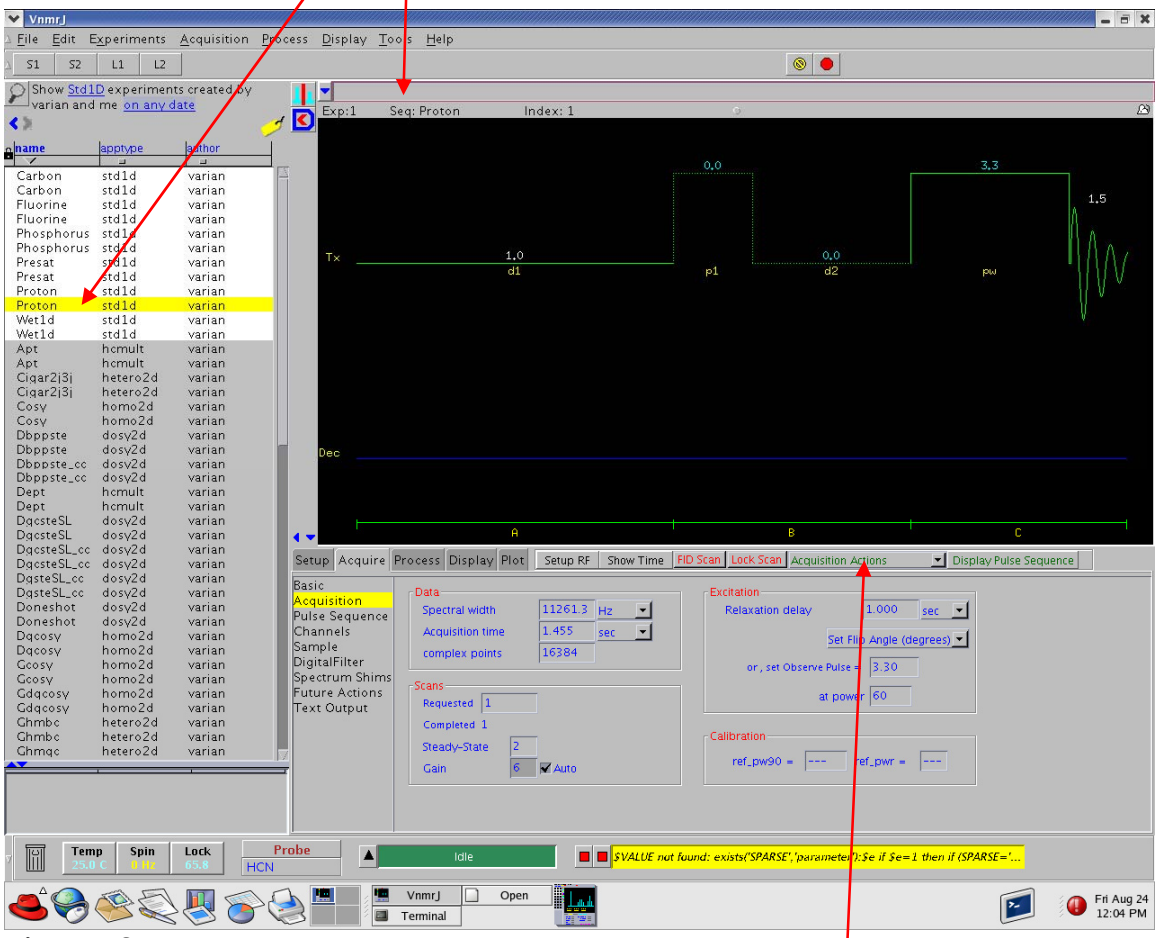



**Figure 18**

This will load the standard proton protocol (pulseprogram) and the appropriate parameters and their values into the workspace exp1. Go to **Setup->Acquisition** and **Setup->Pulse Sequence** to review parameters. Click **Acquisition Action** and select **Star Acquisition** from the drop-down menu to acquire data.

## 3.2 Routine Processing

The spectrum should be processed and displayed automatically. If the integral is displayed, click a few times on **the integral icon** until the integrals are turned off.

If spectrum is not displayed automatically, go to **Process-> Process**.
Click on ***With weighting*** or ***Without weighting*** under **Transform** to do Fourier Transform with or without a weighting function. Before you do this, make sure **FT Size** is at least twice the number of points. Type **aph** at the VnmrJ command line to do automatic phase correction.

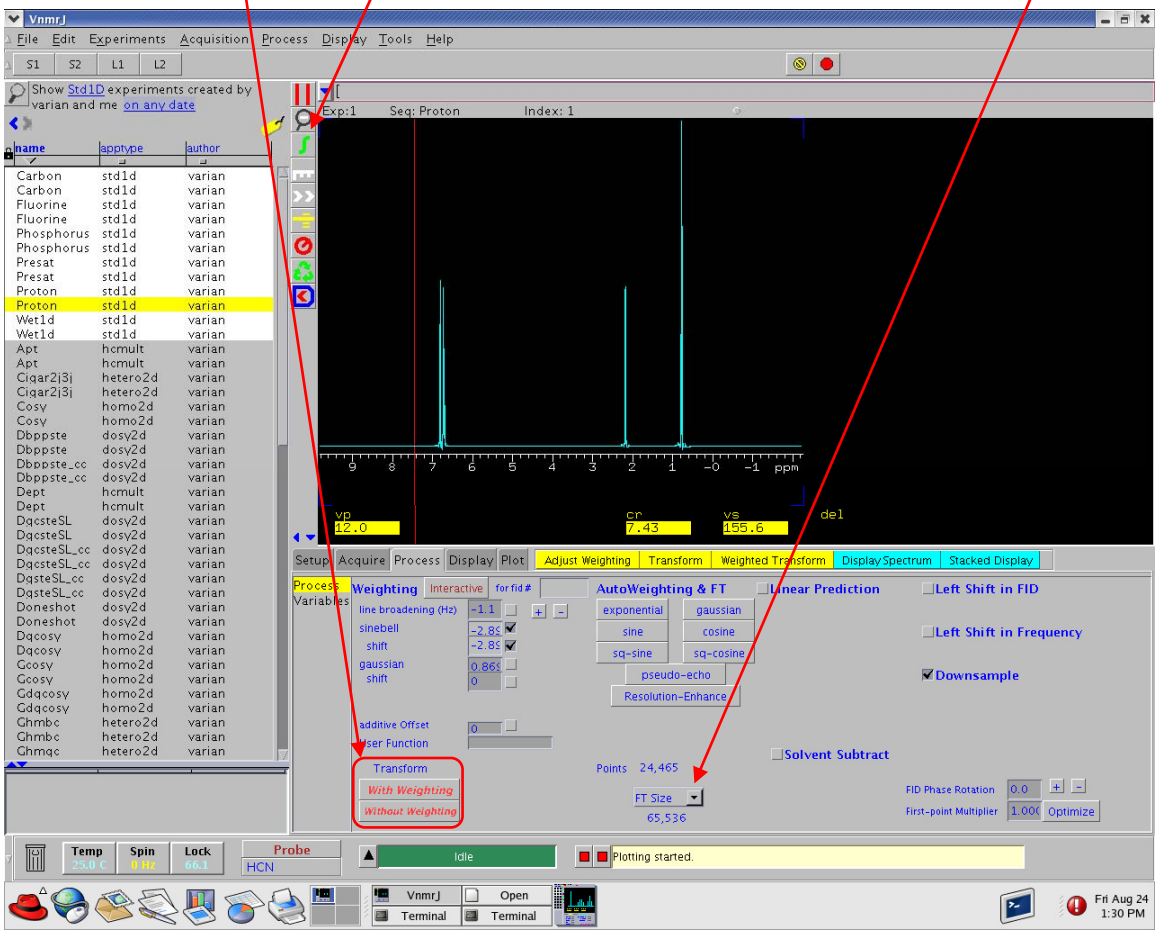



**Figure 19**

If spectrum is not well phased using autophase, manually phase:
Click on phasing tool icon (figure below). Mouse buttons will now have new functions.

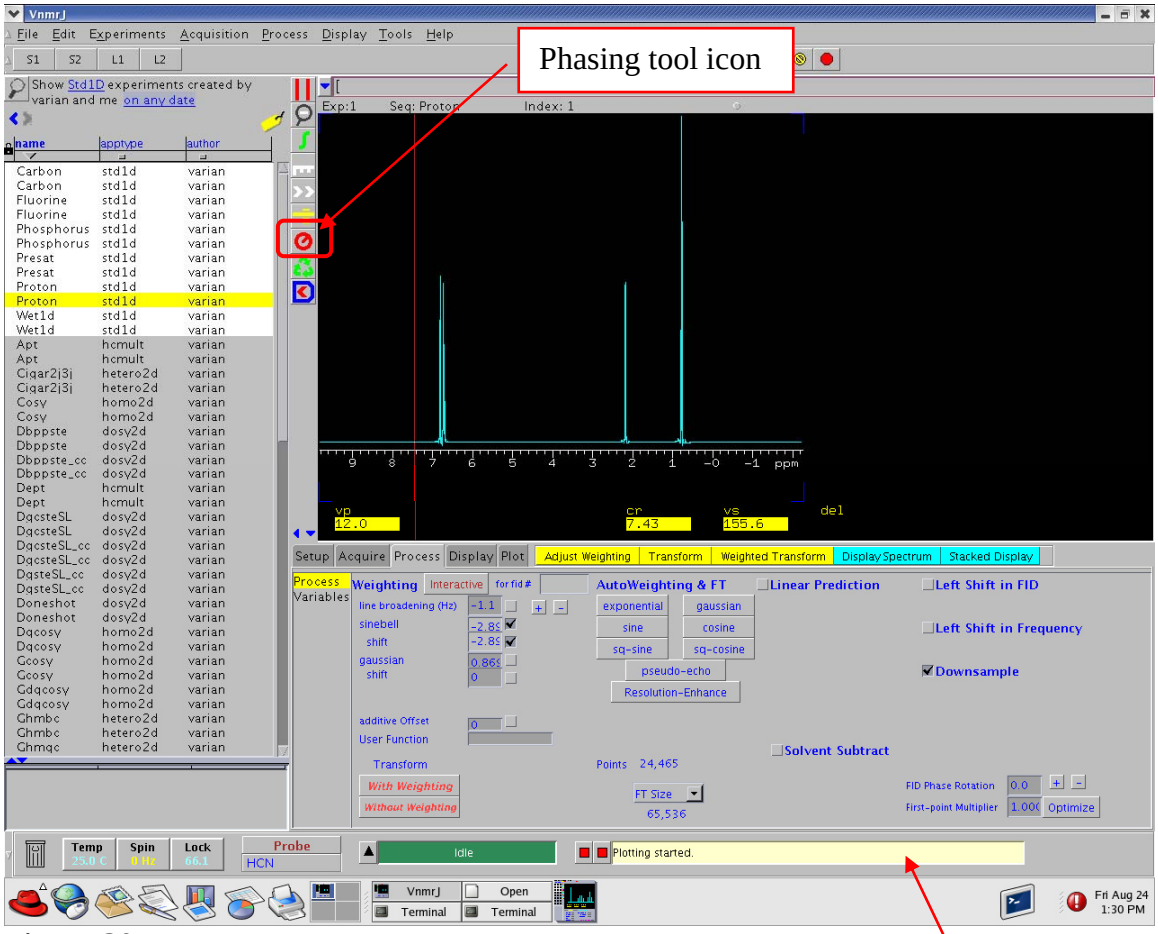



**Figure 20**

Click left mouse button on a peak on right of spectrum.
Now mouse buttons are: LEFT – coarse phase control, RIGHT- fine phase control, CENTER-vertical scale.
Sliding mouse while holding left or right button will adjust phase on the selected peak. This is the zero-order phase that is applied equally across the spectrum (type **rp**? At the command line and its value will be displayed at the bottom of the screen on the **message display window**).
Now move mouse to peaks at left of spectrum and click left button.
Sliding mouse while holding left or right button will adjust phase in this region of the spectrum while leaving the initial peak as before. This is the first order phase correction (**lp**).

**If right peak is slightly out of phase, re-click and re-adjust phase.**

3.3 1D Plotting.

## 3.3 Routine Plotting

Open the **Display** panel and setup preferences such as axis label (whether ppm or Hz) etc the go the **Plot->1D Plot** panel and click on **Automatic Plot Now** for a standard plot.

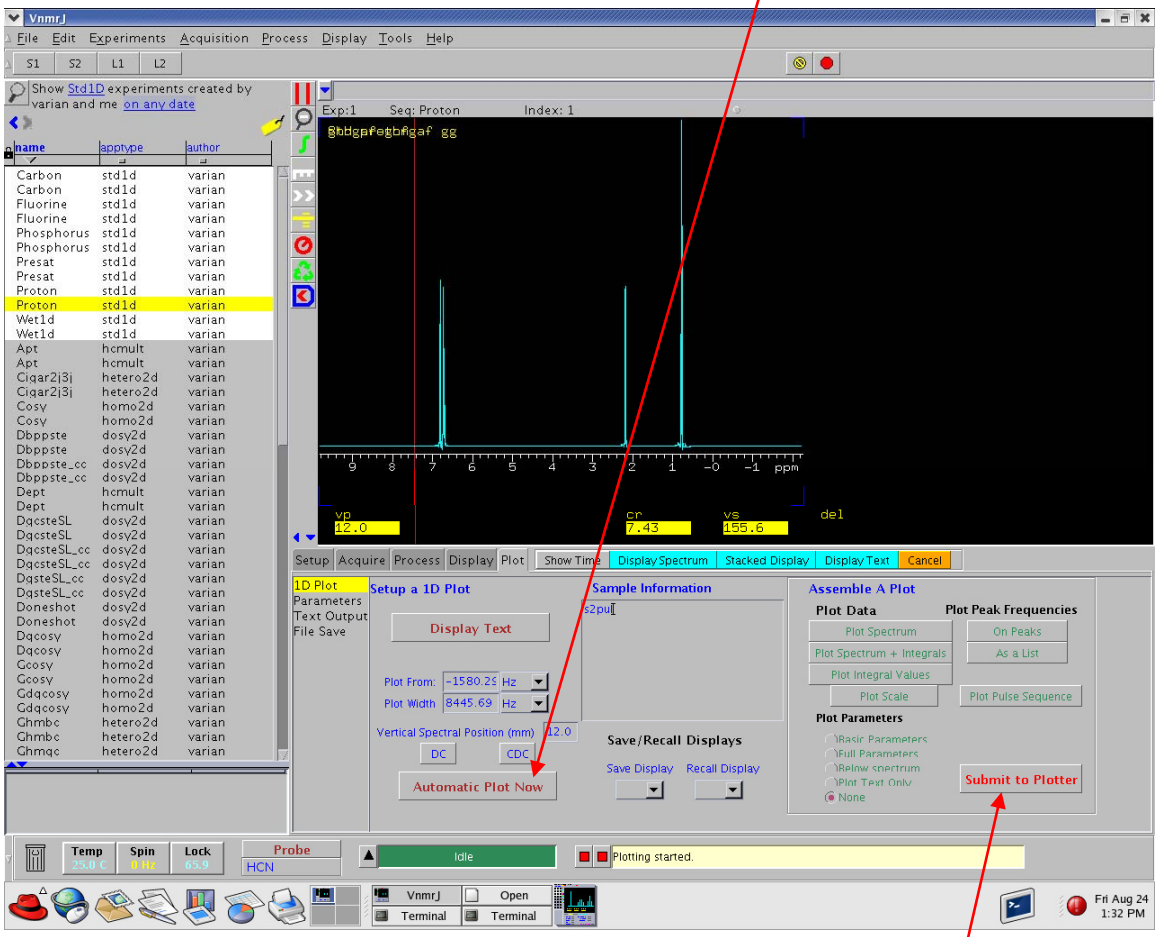



**Figure 21**

Custom plots can be made by choosing plot elements (spectrum, scale, parameters, etc.) and then sending the created plot to the printer by clicking the **Submit to Plotter** button.

## 3.4 Saving the data

The dataset and parameter files are automatically written to the disk in the workspace exp# under generic names – they will be overwritten at the start of the next acquisition. To save the data to a permanent file name, go to the command line and type **svf** and click enter. Write desired name at the prompt and hit return. The path to the place where the file is saved can be found by typing **pwd (**for publish working directory**)** at the command line.

## 3.5 Optimizing Acquisition parameters.

### 3.5.1 Evaluating signal to noise and lineshape.

Get two cursors; left button for left cursor, right button for both.

Expand around some peaks for a close look (click on magnifying glass) .

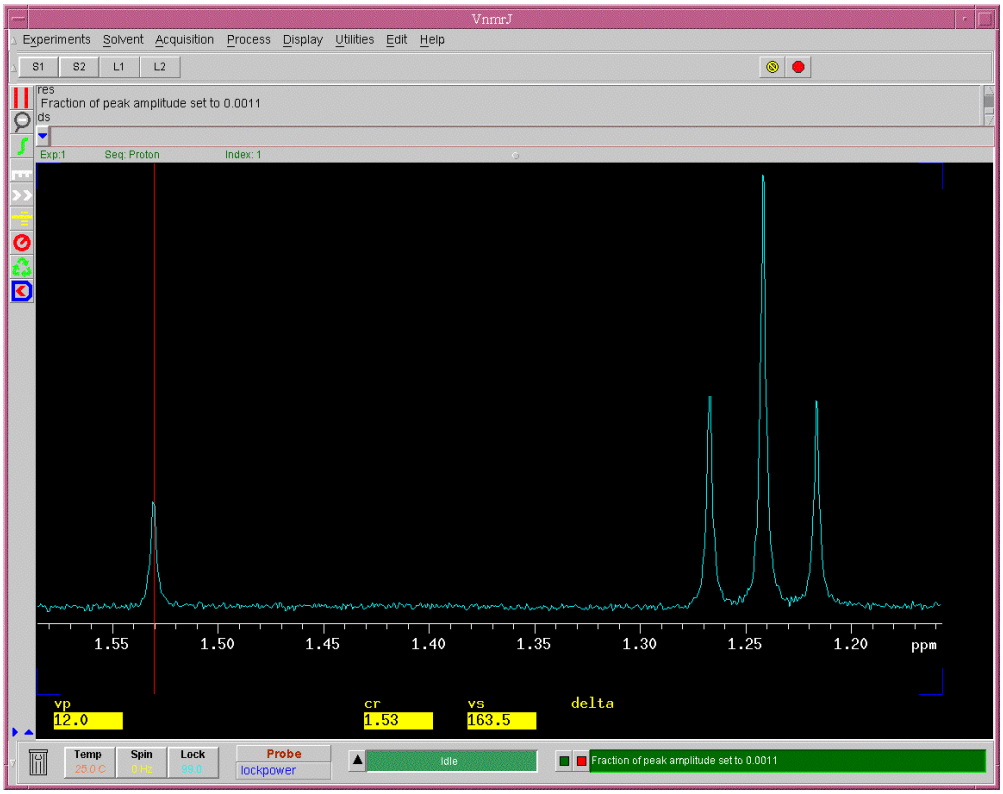



**Figure 22**

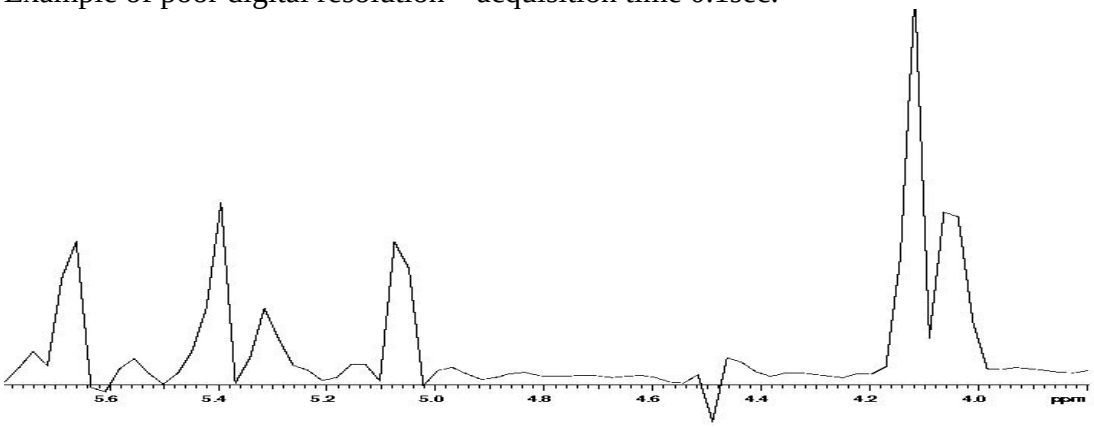
Assess the spectrum visually for symmetric, narrow, smooth lines with sufficient separation and resolution of multiplets. If the lineshape is poor, re-shim.

### 3.5.2 Digital resolution

Go to Aquire-Acquisition panel.
The digital resolution is defined as follows:
Hz per point = spectral width (Hz) / number of acquired complex points

It is usually expressed as Acquisition time; i.e. time when the receiver is turned on.
Acquisition time (sec) = number of acquired complex points/ spectral width.
The default acquisition time (1-2 sec) is more than adequate for high digital resolution.
The default processed data size or “Transform size” in Proc-Default should be equal to or about twice the acquired points.
If the spectrum is not smooth check that acquisition time is > 0.5 sec. and that the processed data size is equal to or greater than the acquired data size.

Example of poor digital resolution – acquisition time 0.1sec.




Standard values- acquisition time is ~ 2 sec.

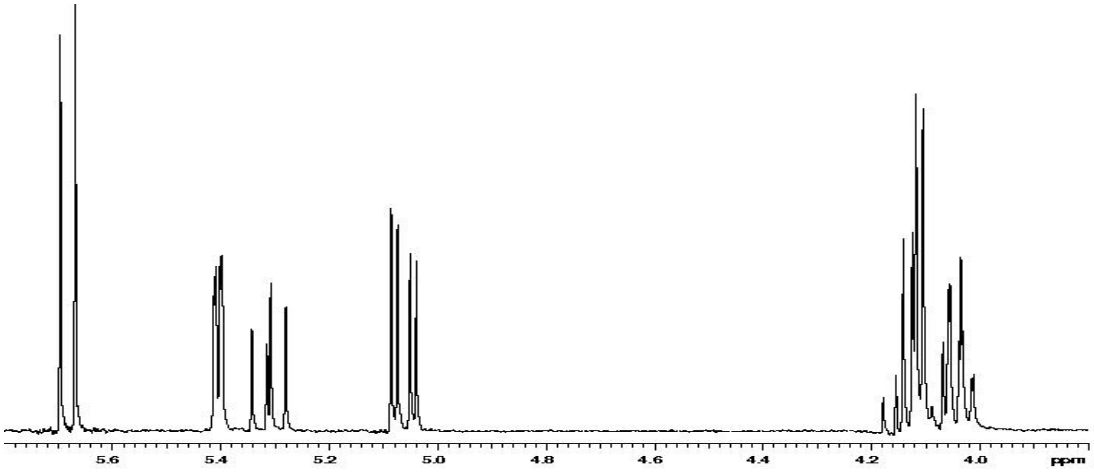



**Figure 23**

### 3.5.3 Optimize Spectral width

This is not necessary for a 1D proton, but is necessary for setting up 2D experiments.

Using the cursors (see above) center the full spectrum leaving some region of flat baseline on either side.
Go to **Display->Cursors/Integration** panel and click on **Reset spectral width** or type **movesw** at the command line and press enter. This changes both the center of the spectrum and the spectral width.
Re-acquire the spectrum.

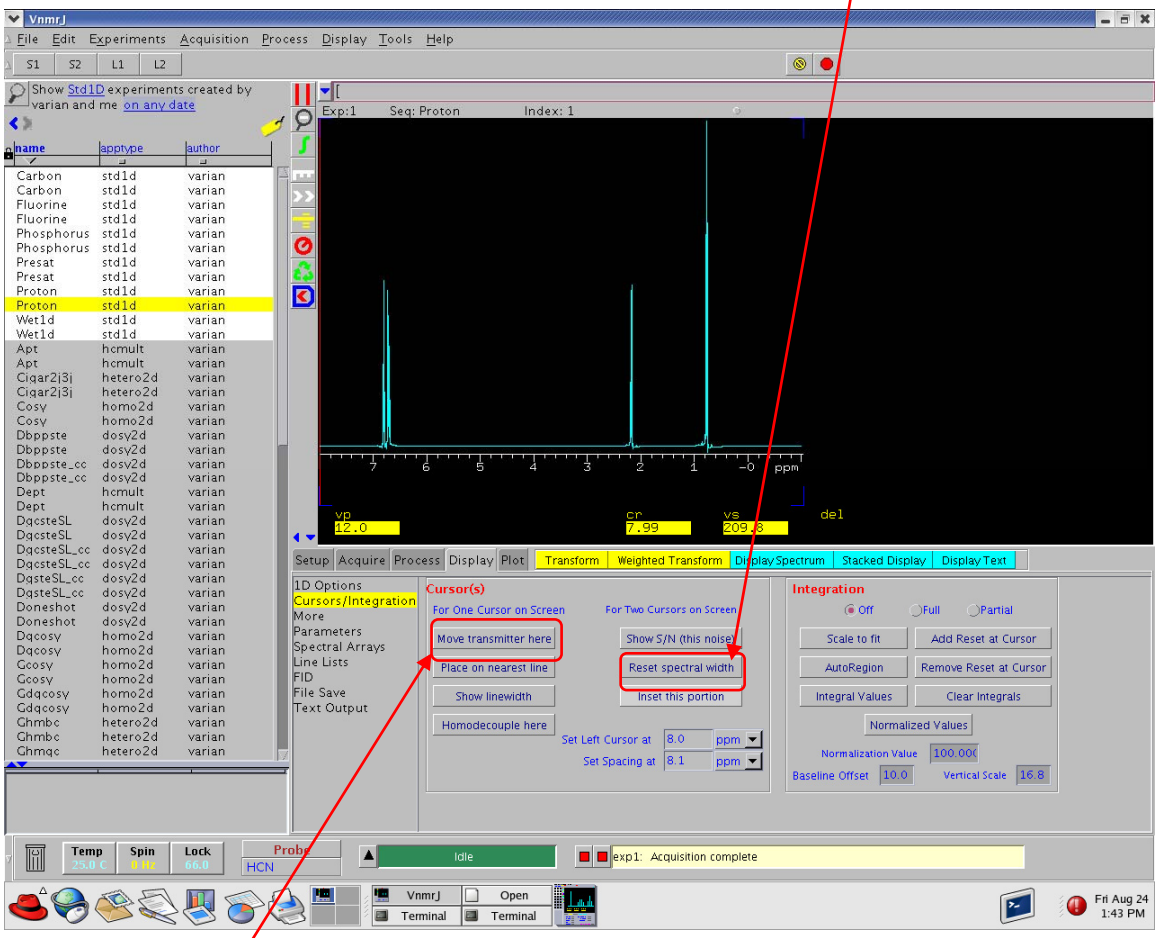


Figure 24

If you want to move the center of your spectrum (the transmitter position) place a single cursor in a position where you want your transmitter position to be and click on **Move transmitter here.**

### 3.5.4 Optimizing pulse width

*Method1: Estimate a 180° pulse.*

Starting from standard Proton setup, collect a phased 1D with a single scan and no steady-state scans. Also turn off the automatic Receiver Gain. (Set these values from Acquire-Acquisition). Observe peaks that are close to the center of the full spectral window, and ensure they are phased correctly.

In **Acquire-Pulse Sequence**, change Observe Pulse to twice the Calibration: pw90 value and type **ga** at the command line.

Residual peaks in spectrum should look noisy with approximately equal positive and negative intensity (like a dispersion spectrum, or like 90° out of phase).

If the peaks are too positive, then increase Observe Pulse value by 1 μsec or so. If they are too negative, decrease Observe Pulse. Re-acquire.

A little experience will allow you to quickly zoom in on a good 180° pulse. The correct 90° pulse is then half of this value.

*Method 2: Setting up an Array of pw.*

Varian software allows easy setup of arrayed parameters. Starting the acquistion will then collect as many spectra as are in the array. These can simultaneously be processed and displayed together for comparison. First acquire a standard 1D proton spectrum with default parameters. You only need enough signal-to-noise to see some peaks, so you can reduce the number of scans to 1.

Click on **Acquisition Action** and select **Array parameter** from the dropdown menu.

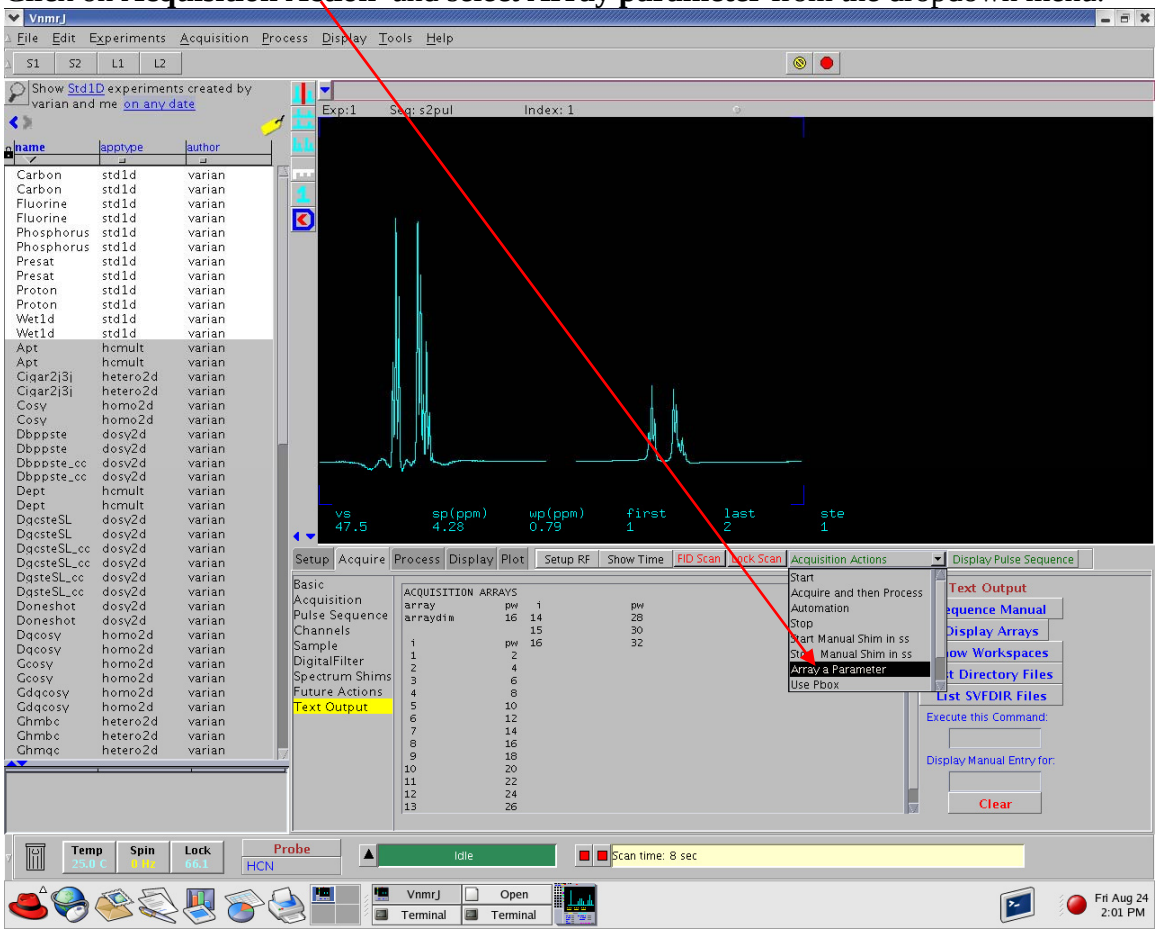



**Figure 25**

Enter **pw** for parameter name in Array Parameter panel and enter the array size and either increment value or last value.

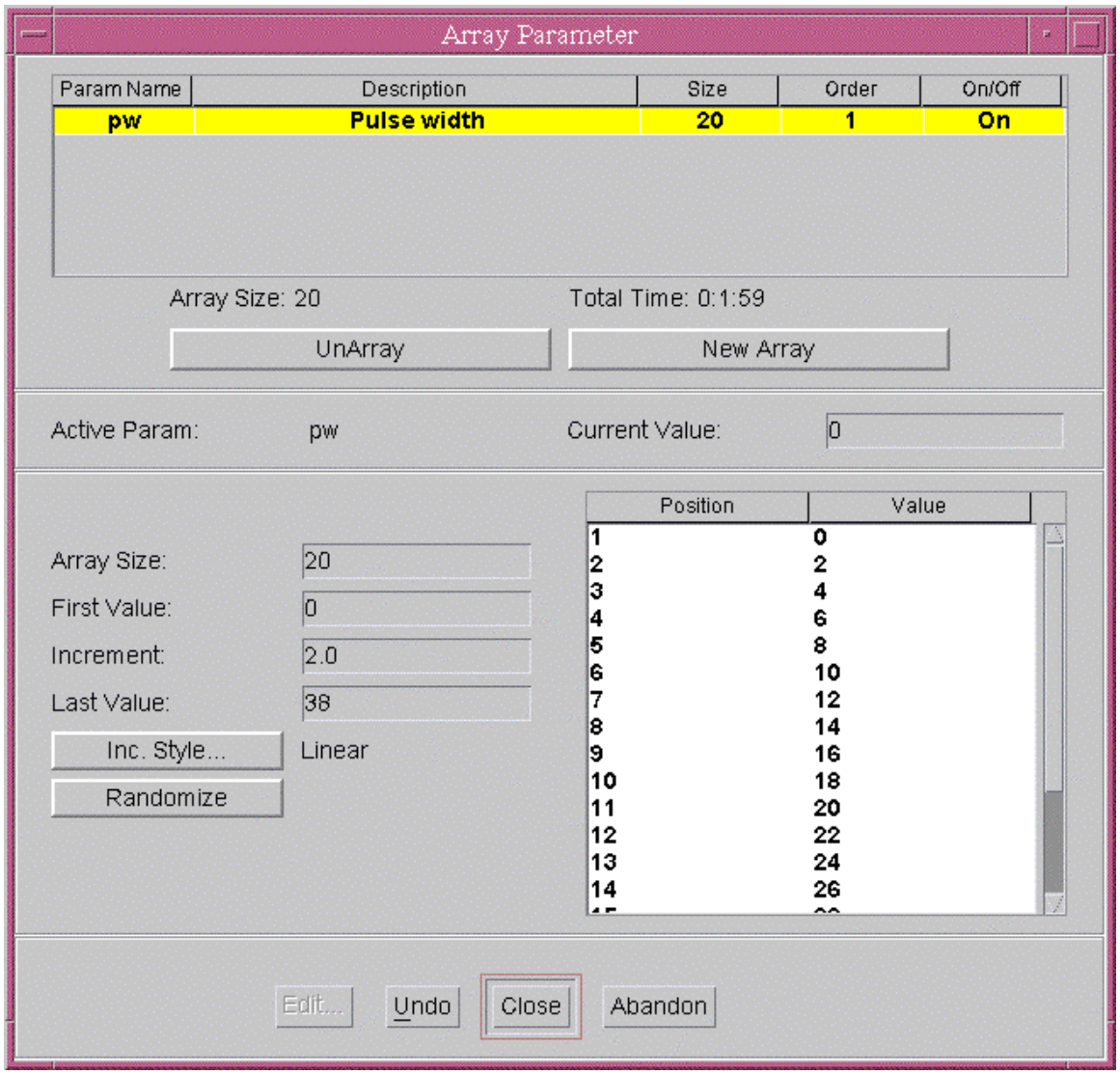



**Figure 26**

For example, the entries in Figure 26 set up a series of 1pulse experiments where the pulse width (pw) is varied from 0 μsec to 38 μsec in 2 μsec steps. Once the array is set, type **ga** at the command line to start acquisition. All 38 spectra will be acquired in a single file.

Go to **Display-> Spectral Array** and click **Transform**, then click on the box for **Display Stacked Horizontally** to see all spectra horizontally. Better, type **dssh dssl** at the command line to see the same spectra labeled with array number as shown bellow.

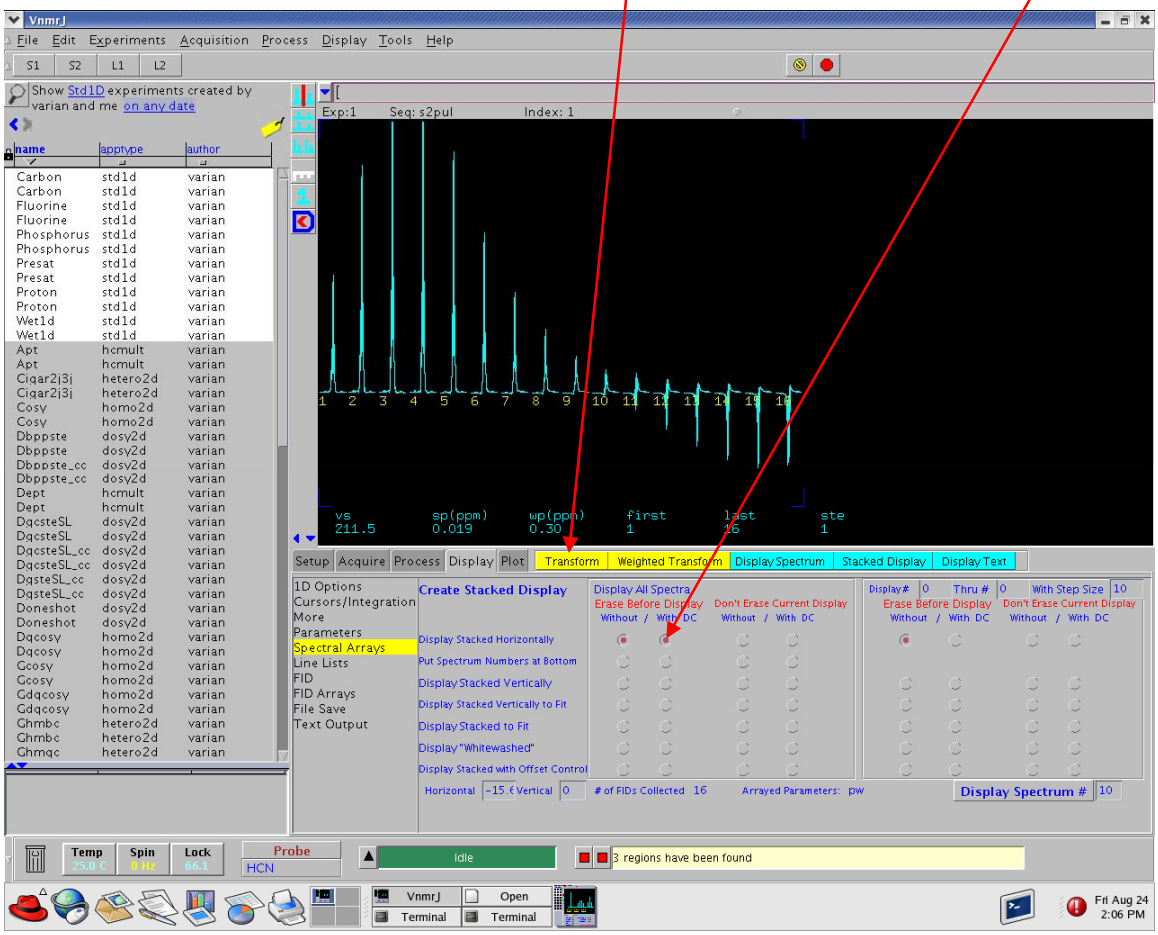



**Figure 27**

The 90° pulse is the pw value of the maximum signal, the 180° pulse is at the first crossover point (zero signal). Refer to the Array window for values.

## 3.6 Optimize processing parameters.

### 3.6.1 Window Functions.

Go to **Process->Process** and choose from the **Autoweighting & FT**; click on desired function, change Weight Parameters and Transform to observe effect.

To examine various functions interactively, first expand around a region of peaks. Go to **Process-Process** and click on **Interactive.**

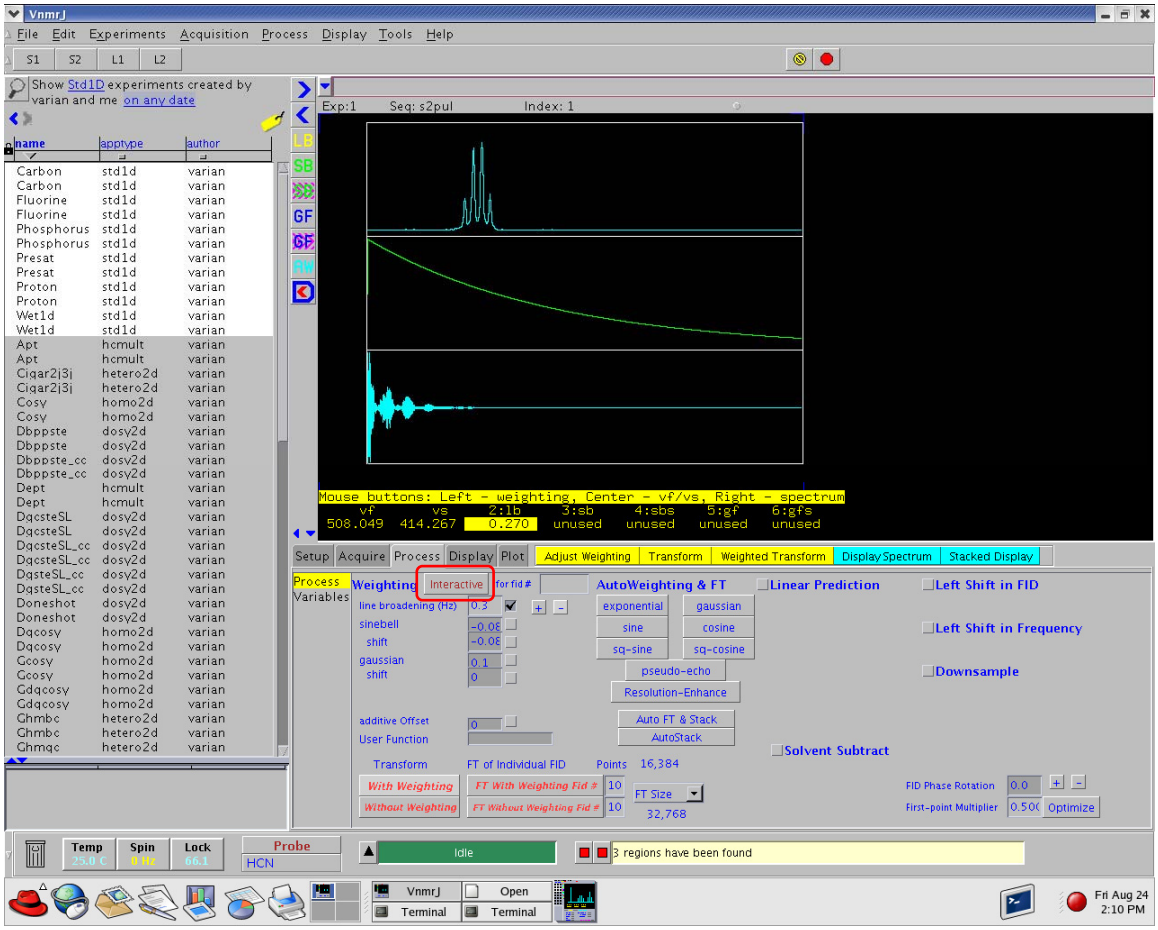



**Figure 28**

Choose a common window function (exponential LB; Gaussian GF: or sinebell SB).
Click above or below the center green line to change the values of the function.
The spectrum in the top panel will change accordingly.

### 3.6.3 Baseline correction

To make sure spectrum is level, and is at the bottom of the display, the baseline should be corrected. This is necessary for accurate integration.
Go to **Display**, click on **DC Correct** or type **dc** at the command line and press enter. This provides a basic zero-order correction.

### 3.7.1 Referencing

Spectra are referenced by default from the solvent chemical shift. For manual referencing to an internal peak, place cursor on the peak and type **nl** at the command line and press enter. Then go to **Display->More** and enter the reference frequency (ppm/Hertz) and click on **Reference Now**. You can do the same thing from the command line by typing **rl(0.000p)** to set the reference line to 0.000 ppm.

### 3.7.2 peak picking

Make sure baseline is flat with DC Correct – see above.
Go to **Display-Line lists**.
Set a peak level **threshold**, click **Display Line list**.

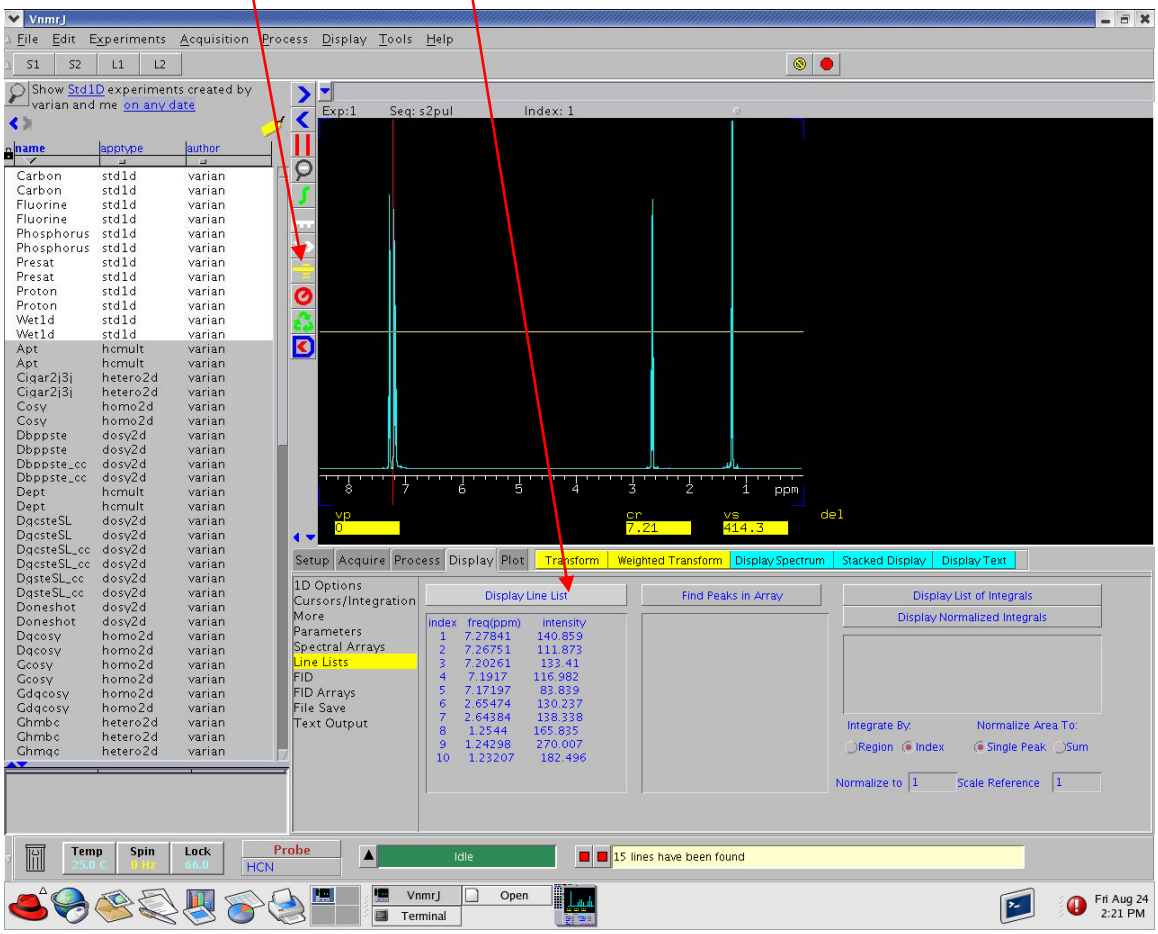



**Figure 29**

Coupling constants in Hz can be determined by expanding the region around a multiplet, placing the two cursors on peaks in the multiplet and typing **delta** at the command line. Alternately one can change axis to hertz (**Display**) and read the delta value directly from the screen display. Changing the axis and creating a line list can be used to calculate the difference in Hz between two peaks in a multiplet.

### 3.7.3 Integration.

Baseline must be flat for quantitative integrals.
Go to **Display->Curors/Integration**.
For simple well defined peaks, click on **AutoRegion** to select integrals.
For selecting integral regions manually, click on integral tool, which will spawn additional integral icons.
Click on **2nd integral icon**; the left mouse button will now choose integral reset points.

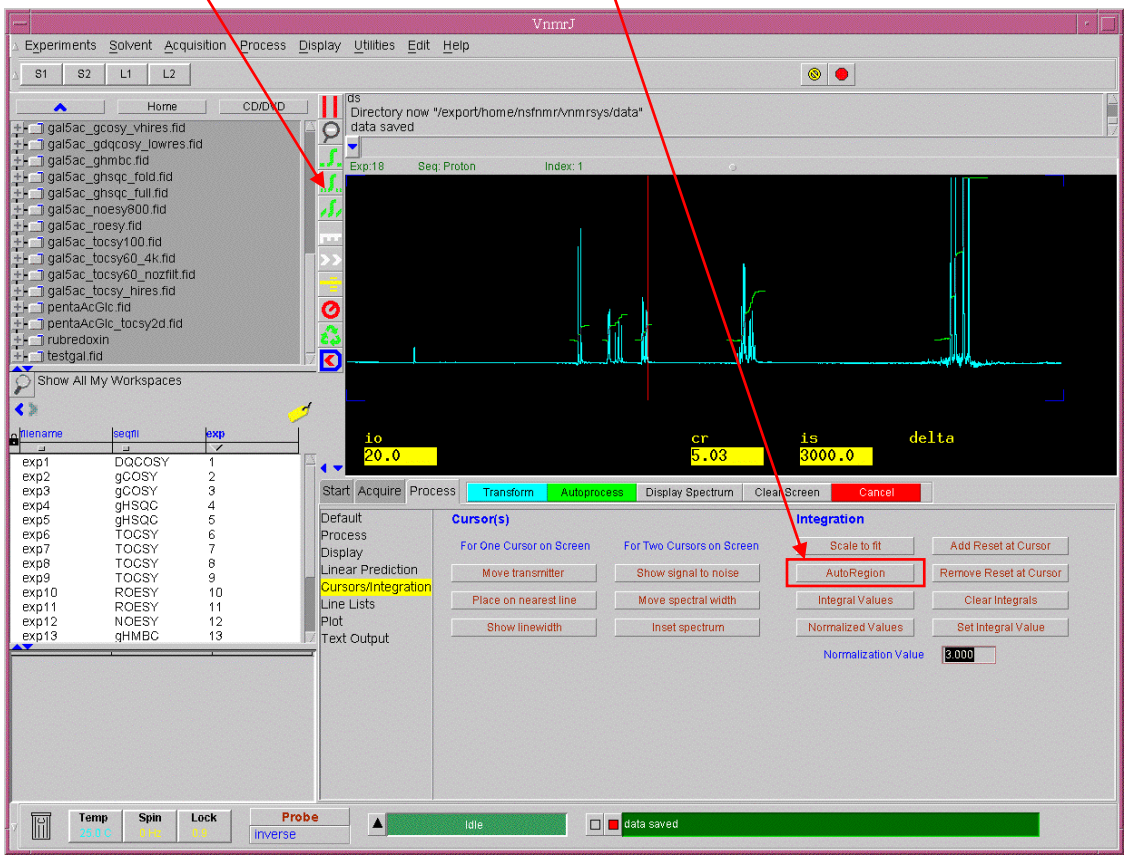



**Figure 30**

Click left mouth button at the left end of a peak to be integrated then click the left mouth button just to the right of the peak. This defines the integral region for that peak. Do the same for all peaks or regions of interest. To normalize the integral type **setint(int, value)** where int is the integral region (left to right starting from 1) and value is the value we want to normalize it to. For example in if I want to set the first integral to 12 fro 12 protons I will type **setint(1,12)** at the command line and press enter and all integrals will be scaled accordingly.

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## 3.8 Presaturation.

Presaturation is the easiest way of suppressing a large signal, usually solvent peak, by irradiating it with a long, low-power pulse prior to the excitation pulse.

Select Presat from the Locator Protocol list. You will need to aquire a preliminary spectrum, so set Scans Requested to 1 and acquire.

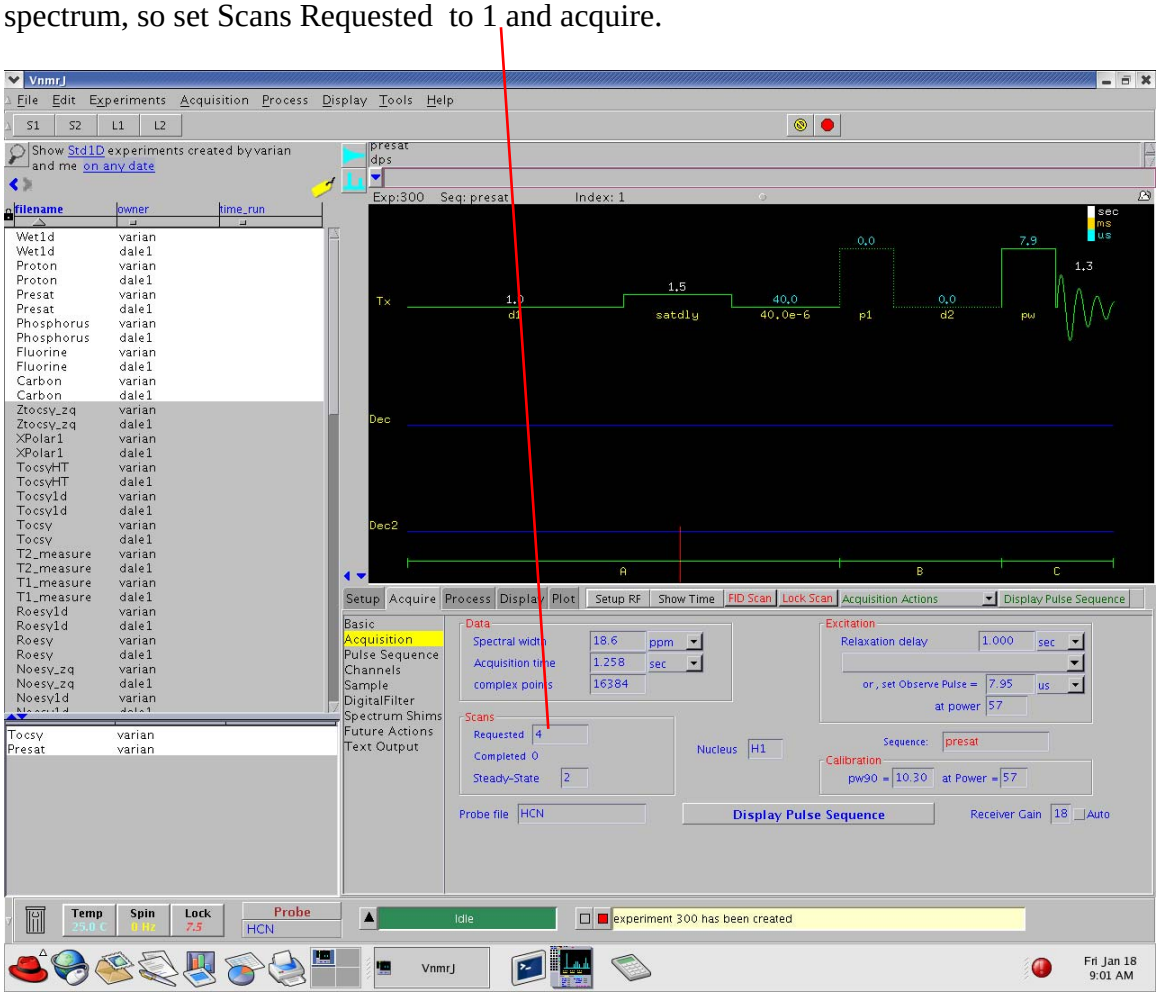


**Figure 30**
Transform and phase. Place single cursor on the peak to be irradiated and type **setpresat**. This will set the saturation frequency to that peak.

Reset the parameters (number of scans, spectral width, etc.) and re-acquire.