

CAMCOR NMR Spectroscopy Facility

Getting Started with VnmrJ 4.2A and OpenVnmrJ 1.1A

<u>Instrument</u>	<u>Location</u>	<u>Probe</u>
Varian Inova-300 “South”	Kla-344	^1H , ^{31}P , ^{19}F (“4-nucleus”)
Varian Inova-500	Kla-344	AutoXDB (any nucleus via “ProTune”)
Varian Mercury-300	Kla-60	^1H , ^{13}C , ^{31}P , ^{19}F (“4-nucleus”)

NOTE: The Kla-60 NMR lab shares space with the undergraduate teaching labs. Safety protocol mandates the following dress code: long pants, closed-toe shoes, and safety glasses/goggles.

FIRST TIME NOTES:

The **first time** you use your account on **each** spectrometer configure your **VnmrJ** workspace:

- Load standard proton parameters: click on **Experiments** → **Proton**
Note: this is very important to properly populate the workspace with valid parameters.
- The command line will be just above the spectrum display window, pull it down, lowering the margin to make space for displaying errors, old command entries, etc.
- Check the printer settings: **File** → **Printers...**
set **printer** to “ps” or “ps[b+w]”
- Create additional workspace(s). exp2, exp3, etc.: click on **File** → **New workspace**

Workspaces:

The user may have several “experiments” (a.k.a. “workspaces”) open at once (only one active for acquisition). To move between experiments type **jexp1**, **jexp2** **jexp3**, etc.

The current Exp: # is shown in the blue band at the top of the spectrum window.

Default Parameters:

- Load default nucleus/acquisition parameters from top-bar: **Experiments**
- Set the solvent, e.g. CDCl_3 , using the **Start** → **Sample info** tab in the lower-right half of the VnmrJ window.
- Go to the **Start** → **Lock** tab. Check for current lock phase value posted on the white board next to the console. The lock phase is different for each spectrometer and varies over time.
- Optional: re-load the standard shims, **Start** → **Shim** → **Read default shims** .
or type on the command line **rts('std5') su** [enter]
- If you get weird error messages from doing the above steps try closing & restarting VnmrJ
- All samples are to be run with **sample spinning turned OFF!** (leave “Spin” unchecked)

TUNING/LOCKING/SHIMMING

- Go to the **Start** tab to eject the sample currently in the magnet and load your sample. Be sure to thoroughly clean the outside of your sample tube and carefully set the tube position with the gold-colored Varian sample depth gauge. Be sure the eject air supports the sample before you let go!
- For the 300 MHz spectrometers, the probe is normally pre-tuned to fixed frequencies (“4-nucleus”). For the 500 MHz, probe tuning is required on each type of sample (affected by solvent, concentration, volume). See notes at the end of this guide.

- In the **Lock** tab, click **Find Z0** to automatically engage the ^2H locking. (this takes a little time... you will see various messages as it progresses). Check that the **lock phase** is correctly set as described above, under **Default Parameters**.

- Click **Gradient Shim** -- takes a couple of minutes to finish. This automatically shims the Z-series of shims. The gradient shimming will do a good job adjusting the **Z** parameters.

Hint: For CDCl_3 samples at 300 MHz manual shimming of **Z1C** and **Z2C** (± 1 increments) will usually be quicker and better than automated gradient shimming due to low ^2H sensitivity.

- On the 500 MHz you may also need to manually adjust the following: **X1, Y1, XZ, YZ, XY, X2Y2**. These are accessed through via the **Start** -> **Shim** tabs. Increment size of ± 10 is recommended; to toggle through increment sizes, click the shim buttons with the *middle* mouse button. The X & Y shims must be adjusted with sample-spinning turned off.

BASIC PROTON SPECTRUM

- Go to the **Acquire** tab. Check acquisition parameters, as needed. You may want to use **nt=1** for a test scan.
- Acquire a standard ^1H , with **nt=8** scans. Click on **Go**.
- Phase as needed (**aph0, aph**, or use interactive phasing (tool on right-side bar.))
- Type "**vsadj**" to adjust vertical scale, and "**cz**" to clear integral resets.
- Integrate (ask for help with integration, if necessary.) Normalize integrals to one peak of your choice. In the plot menu chose "plot normalized integrals horizontally".
- Plot the ^1H spectrum: on the **Process** tab, click **Plot**
 - Select the graphics and lists that you would like to include in your plot
 - If printing all parameters, type **wc=160** to avoid spectrum and parameters overlapping
 - Click **Preview** to view in Adobe Reader.
 - To print, select "Print 1 page" and click **Print**.

WHEN YOU ARE DONE:

- Replace the CDCl_3 standard in the magnet
- Exit VnmrJ
- Log out of the Linux desktop. Be sure to "confirm logout" (do NOT use "switch user.")
- Be sure that you have signed the log book.

PROBE TUNING (for 500 only)

Tune the probe (after you've inserted your sample):

- In the **Start** tab, click **Auto tune** then choose, e.g., **Quick Tune C13** in the **Tune Probe** pop-up menu (upper left of screen). The *protunegui* window transiently appears during the tuning progresses. When tuning each nucleus finishes you should note the "**protune_au done**" message in bottom status bar.
- Tune the X-nucleus first (e.g. ^{13}C),
- Then the ^1H decoupling channel, if applicable, with **Quick Tune H1**.
- Close the **Tune Probe** menu.