CAMCOR NMR Spectroscopy Facility – Getting Started with VnmrJ

Instrument	Location	Probe
Varian Inova-300 "South"	Kla-344	^{1}H, ^{13}C, ^{31}P, ^{19}F ("4-nucleus")
Varian Inova-300 "North"	Kla-344	^{1}H, ^{11}B, ^{31}P, ^{19}F ("4-nucleus")
Varian Inova-500	Kla-344	AutoXDB (any nucleus via "ProTune")
Varian Mercury-300	Kla-60	^{1}H, ^{13}C, ^{31}P, ^{19}F ("4-nucleus")
Bruker Avance III-HD-600	Kla-60	Prodigy cryoprobe (broadband), CP/MAS *(600 upgrade installation: summer 2012)*

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.*
- Enable the VnmrJ Command Line from the View pull-down menu (top).
- Command line appears appears just above the spectrum display window,
- next to ▼blue triangle.
- Check the printer settings: File → Printers...
  set ***plotter*** to "ps[b+w]" and ***printer*** to "text[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.* CDCl3 using the Start → Standard tabs.
- Optional: re-load the standard shims, Start → Shim → Read default shims .
  or type on the command-line ***rts('std5')*** ***su*** **[enter]**
- 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.
- If you get weird error messages from doing the above steps try closing & restarting VnmrJ

TUNING/LOCKING/SHIMMING

- Go to the Start → Standard tabs in the lower-right half of the VnmrJ window 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 sample depth gauge.

- For the 300 MHz spectrometers the probe is normally pre-tuned to fixed frequencies ("4-nucleus"). For 500 & 600 MHz probe tuning is required on each type of sample (affected by solvent, concentration, volume). See notes at the end of this guide.

• Click the "Find Z0" button to automatically engage the ^{2}H locking.
(this takes a little time... you will see various messages as it progresses). Check that the **lock phase** is correctly set (current value should be posted on white board next to console). It is different on each console and may vary with time.

• Click Gradient Shim button-- 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.

On the 500 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; click the *middle* mouse button when pointing at each shim button to toggle through increment sizes. The X & Y shims must be adjusted with sample-spinning turned off.

BASIC PROTON SPECTRUM

• Click on the gray Acquire tab (not the Acquire *action* button). Check acquisition parameters, as needed. You may want to use ***nt=1*** for a test scan.
• Acquire a standard ^{1}H, with ***nt=8*** scans. Click on Acquire.
• 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
- Click Plot Spectrum
- Click Plot Parameters
- Click Plot Integrals
- Click Plot Spectrum Scale
- Click **Plot Preview**. Adobe Reader will open.
  You can send to the printer and/or save as a PDF file.

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"
• Be sure that you have signed the log book.

PROBE TUNING ***(for (500only)***

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

• In the Start → Standard menu, click on Tune then, *e.g.*, choose 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. 13C), then the 1H decoupling channel, if applicable.
• Then Quick Tune H1. Close the **Tune Probe** menu.