BARE BONES GUIDE to the Operation of the
Varian Inova-400,500 and VXRs-300
NMR Instruments

This guide is intended to accompany proper training and formal check-out by the NMR Facility Director, or the
Director’s Designee. You must be OK’d by Facility staff prior to using the instrument. This guide covers the
most basic operational details for acquiring and storing routine 1-dimensional NMR spectra for the most common
(routine) nuclei: $^1H, ^{13}C, ^{31}P,$ and $^{19}F$.

Other guides are (or will be) available for processing/plotting, performing more advanced experiments or for
observation of other nuclei.

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Conventions in this manual:

**Boldface** text indicates commands to be typed at the computer

<angle brackets> are used to designate a key to be pressed (i.e. <Ret> for Return/Enter)

[square brackets] designate an icon/button in the VNMR menu to be clicked

**Mouse Conventions:** *click*, by default, refers to the Left Mouse Button.
- **LMB** will be used to designate the Left Mouse Button
- **MMB** will be used to designate the Middle Mouse Button
- **RMB** will be used to designate the Right Mouse Button
Sometimes you will need to *hold*, rather than *click* the mouse button. This means that you should
press and hold the button down throughout the operation.

1) **SIGN IN** the Log-Computer *(Excel Spreadsheet)* immediately before you start… follow instructions provided.
2) If the screensaver is active, *click* a mouse button, or hit <Ret>, then type the password to unlock the screen.
3) **Switch to your Data Directory**
   a) *Clicking* on [Main Menu] then [rgroups] in the menu, then select your group directory (LMB click), then click on
      [Set Directory].
   b) *Click* on your directory name (so that it is highlighted), then click on [Set Directory].
   c) You should notice the message displayed at the top of the screen, which indicates that the current directory is your
directory.
4) **Remove the Reference sample, INSERT your sample and LOCK**
   a) Type e <Ret> to eject the sample, and carefully remove the sample from the top of the magnet, remove the
   reference tube from the spinner turbine.
   b) WIPE your NMR tube thoroughly. with a Kim-Wipe, slightly wet the Kim-wipe with alcohol if necessary. Be
   sure the NMR tube is clean.
   c) Put your sample in a spinner-turbine, and then wipe the tube again! (before setting the sample depth)
   d) Set the proper sample depth.
      i) Use the depth gauge by placing the tube in the turbine, place the turbine on the gauge, and push the sample
down until it touches bottom.
      ii) If the sample is "shorter" than normal, be sure that the sample is centered above/below the observe coil
limits (use the dashed-rectangle on the gauge, or the decal on the wall to check).
   e) Double-check the sample depth, then carefully place the sample in the top of the magnet.
   f) Type i <Ret> to insert your sample.
   g) **OPTIONAL:** You can type reset <Ret> to recall the Standard Shims for the Instrument/Probe to be sure you
are starting with decent shim values. WAIT for the **Beep** and "Setup Complete" message!
   h) *Click* on [acqi] to bring up the Lock/Shim panel. (NOTE: if you don't see the button on the top row, try typing
   acqi <Ret> on the keyboard).
i) Click on [Lock] to bring up the lock window.

   Note: The instrument may have locked automatically when you inserted your sample. If so, you can skip to Step (iv) below (setting lock power). Always do steps iv, v, & vi.

   {Note: In these panels, clicking LMB on [+] will decrease/subtract from the parameter value, and clicking RMB on [+] will increase/add to the corresponding value.}

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   ii) Use the CHART by each instrument to pre-set Z0 to approximately the correct value for your solvent. If you see periodic, sinusoidal oscillations, adjust Z0 to minimize the frequency of these oscillations to near zero (straight line).

   iii) Click on lock [Off], and the instrument should lock; however, the Lock Power may be too high.

   iv) Set the lock power to an appropriate value for your solvent (Note the probe sign for CDCl3 (1)).

   (1) Relative to CDCl3, use the following powers for other solvents:

   (a) Acetone/Acetonitrile/Methanol: subtract 15 or more; DMSO, D2O, CD2Cl2, C6D6: subtract at least 10

   If you don’t see the lock signal, raise the lock power using the slider (hold LMB on the slider to move w/ mouse), so you can see the lock signal.

   {Note: In these panels, clicking LMB on [+] will decrease/subtract from the parameter value, and clicking RMB on [+] will increase/add to the corresponding value.}

   v) Adjust the Lock Gain to set the Lock Level ~mid-range (just over 50% is good).

   vi) Check that the lock phase matches the value on the probe-sign (on the wall).

5) SHIM the magnetic field (if you are on the 400 or 500 you will usually only do Z1 & Z2, then gradient shim):

   a) Click on [Shim] to enter the shim routine, make sure the mode for the shim window is [manual].

   b) The objective is to maximize the lock level, using Z1 & Z2 … the actual lock value isn't really important. If the Lock Level goes near 100%, lower the lock gain because you can’t shim if the lock-level is off-scale. Start with [Z1] and [Z2] using the larger (coarser) number [+] buttons first, maximize the level with [Z1], then [Z2] (NOTE: On the 400, use the [Z1C] and [Z2C] [+] numbers first),

   c) On the 400 and 500, Gradient Shimming is recommended.

   You cannot gradient shim with: Toluene, Methanol-d4 or THF-d8, Pyridine-d5.

   If you are going to use Gradient Shimming…, Click [Close] and Proceed to step 6. Note: ALWAYS exit the Acqi window using the [Close] Button.

   d) On the 300…Switch to [automatic] mode, by clicking on the [auto] mode button.

   i) Check to be sure that you see "M>M", and "Z1-Z4" (Z1-Z5 on the 500) on the screen. If not, do step "ii" below.

   ii) Hold the RMB on the [>] button to select "M>M", and "Z1-Z4".

   iii) Then click on [Start], and the button name should change to [stop], which means that auto-shimming is in-progress.

   iv) When done shimming, the button will again change to [Start]. If you can see that little progress is being made by autoleshim, you can click on [Stop]. Generally, if you let autoleshim work for 60 seconds, the results will be satisfactory (assuming that Z1 and Z2 were optimized in step (b).

   e) Close the acqi window by clicking on [Close].

6) Click on [Main Menu], then [Setup], then the appropriate button (do ONE of the following: a, b, or c):

   a) [H1, CDCL3] - routine 1H NMR in CDCl3

   b) [C13,CDCL3] - routine 13C in CDCl3

   c) [Nucleus,Solvent] - routine 1D NMR to observe other nuclei or if using solvents other than CDCl3

   i) First select the nucleus you wish to observe by clicking on the button

   ii) Then select the deuterated solvent you are using for the lock.

   d) Type su <Ret> to setup the hardware, after doing either a), b), or c) above.

7) Gradient Shimming (400 and 500 only)

   a) Most solvents work well, but don’t gradient shim with: Toluene, Methanol-d4 or THF-d8, Pyridine-d5. Try manual shimming or lock-auto-shimming (step 5d above) with these multi-frequency solvents.

   b) Click [Main Menu][Setup][Shim][Gradient Autoshim on Z].

   c) Wait until gradient shimming is completed… You should hear a “beep”, and see “Set Hardware: operation complete”. Also, the instrument should re-lock, and the sample should start spinning again.
8) Set Parameters, before acquiring the spectrum:
 a) Type \texttt{nt = \# <Ret>}, where \texttt{"\#"} is the number of scans (multiple of 4).
   i) 16 to 32 for routine 1H, 512 to infinity for 13C.
 b) Note the value for "bs", this is the block size, and determines how frequently you can examine/process the data
    as the acquisition progresses: i.e. BS=4 means that the data is updated for processing every 4 scans.

9) Begin the Acquisition:
 a) Type \texttt{ga<Ret>} to begin data acquisition.
   i) After you see “BS # Completed”, you can type \texttt{wft<Ret>} to “weight and FFT the data”
   ii) Entering Text
      1) You can enter text for display or plotting, such as a description of the sample, reaction conditions, ...etc.
         a) type \texttt{text(' your text here')} to enter the first line of text.
   b) The spectrum will appear automatically after “NT” scans.
      i) If you selected a large number for NT, but you want to stop it early, type: \texttt{sa('bs')<Ret>}. This stops
         the acquisition at the next multiple of “bs”. NEVER use this command when the acquisition is almost done!!
         If you use this command and the acquisition completes “nt” scans first, you will crash the instrument.
      ii) After the acquisition stops, type \texttt{wft<Ret>}.

10) Phase the Spectrum:
 a) Type \texttt{aph<Ret>} to autophase the spectrum. If this fails, try \texttt{aph0<Ret>}.

11) Save your spectrum (fid) to your directory in \texttt{rgroups}:
 a) To make sure you are in the correct directory, type \texttt{pwd<Ret>}, and check the current directory.
 b) Type \texttt{svf<Ret>} and you will be prompted for your filename.
   i) filenames: please avoid symbol characters (#,!, ?, *, ;, ...etc.), spaces, and multiple dots (.) in filenames.
      Dashes (-) and underscores ( _ ) are the best characters to use as separators. This improves computability
      between different operating systems, especially for backup to CD or Zip disks.
   ii) Enter your filename, followed by \texttt{<Ret>}. Alternatively: you can type \texttt{svf('filename') <Ret>} and save all on one line.
 c) Type \texttt{files <Ret>} (or click [MainMenu][File]) to see your files, make sure it was saved.

12) Type \texttt{e <Ret>} to eject your sample.
 a) If you are done, insert the reference sample (wipe the tube with a KimWipe, and check the sample depth
    before inserting.
 b) If you have another sample, you can remove your sample from the spinner turbine (be careful not to break
    the tube), and insert your next sample into the turbine (wipe the tube, check sample depth).

13) Type \texttt{i <Ret>} to insert the next sample, or the reference sample.

14) Quitting:
 a) When done, the reference sample should be inserted.
 b) Lock and Shim on the reference – (Type \texttt{reset<Ret>} to reset all parameters for the reference sample, and
   WAIT for the \texttt{Beep} and “Setup Complete” message before doing anything else!).
   i) (this lets the next user know that everything is normal, and ready to go). Don't spend much time
      Shimming; rather, just achieve a reasonable lock level.
 c) Type \texttt{cd <Ret>} to return the current directory to \texttt{home}, so that the next user won't accidentally be working in
    your data directory.
 d) If nobody is using the instrument, put the cursor on the CU wallpaper, \textbf{Hold the RMB}, and select \texttt{lockscreen}
    to activate the screensaver.
 e) Log Out in the Logbook, indicating the time used.

15) Data Processing: Instructions for processing, integrating, peak-picking, and plotting are in a separate document.
 All data processing should be done at one of the workstations.