

BARE BONES GUIDE to the Operation of the Varian Inova-400,500 and VXR-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.

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- 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 wipe the tube again!
 - 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).
- i) Click on the lock [Off] button to turn the lock off.
 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.}
- 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 [On], and the instrument should lock; however, the LockPower may be too high.
- iv) Set the lock power back to an appropriate value for your solvent (Note the probe sign for CDCl₃):
 (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
 (2) If the Lock Power is too high, the lock level will oscillate up and down...this is bad.
- 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, 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 & 300, use the [Z1C] and [Z2C] [+/- 1] numbers first**),
- c) **On the 400 and 500, Gradient Shimming is recommended (you can NOT gradient-shim on the 300).**
 On the 400 and 500, 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 *autoshim*, you can *click* on [Stop]. Generally, if you let autoshim 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 **nt = # <Ret>**, where “#” 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 **ga<Ret>** to begin data acquisition.
 - i) After you see “BS # Completed”, you can type **wft<Ret>** to “weight and FFT the data”
 - 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: **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 **wft<Ret>**.
- 10) Phase the Spectrum:
 - a) Type **aph<Ret>** to autophase the spectrum. If this fails, try **aph0<Ret>**.
- 11) Save your spectrum (fid) to your directory in rgroups.
 - a) To make sure you are in the correct directory, type **pwd<Ret>**, and check the current directory.
 - b) Type **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 **<Ret>**.

Alternatively: you can type **svf(‘filename’) <Ret>** and save all on one line.
- 12) Type **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 **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 **reset<Ret>** to reset all parameters for the reference sample, and WAIT for the *Beep* and “Setup Complete” message before doing anything else!)

 - (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 **cd <Ret>** to return the current directory to *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, **Hold the RMB**, and *select [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.