Practical Guide on Varian NMR Systems:

Locking, Shimming, Acquiring, and Processing

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1)	***Login***	At the Sun workstation, inside the *login window*, type: **Your username** <ENTER> **Your password** <ENTER> Wait for the start of the UNIX session and the launch of VNMR program. Obs: All **NMR** commands/parameters are typed in the **VNMR** command line.	
2)	***Inserting the sample***	✓ Type ***e*** <ENTER>	*To eject the standard sample, which is always kept inside the magnet.*
		✓ Replace the *standard sample* **by** *your sample* ***into the spinner***	*Adjust sample's position with the gage*
		✓ Insert the spinner ***with*** your sample into the top of the magnet.	
		✓ Type ***i*** <ENTER>	*To slowly descend your sample into the probe inside the magnet.*
3)	***Locking on the deuterated solvent***	Automatic locking (NMR-300, 400 only) ✓ Type ***setlk ('****solvent'****)*** <ENTER> ✓ Type ***su*** <ENTER> to automatically lock the sample. ***setlk*** *will lock the sample in a few seconds.* *Example:* ***setlk('****cdcl3****') su*** *<ENTER>.* *Lock parameters such as z0, lockpower, and lockgain are called by* ***setlk***	*Manual locking (all spectrometers):* ✓ *Keep the sample* ***static.*** ✓ *Click on the* ***Acqi*** *button.* ✓ *Click on Lock* ***OFF****.* ✓ *Change Z0 in ±4 (or ±16) steps until a flat signal is found* ✓ *Optimize its intensity by changing the Lockphase in ±1 steps* ✓ *Click on Lock* ***ON*** *to enable the lock circuit.* *The message* ***LOCKED*** *in green is displayed at the bottom left of the graphics display*
4)	***Shimming***	Automatic shimming (NMR-300, 400 only) ✓ Type ***gmapshim*** <ENTER> ▪ *During ^{2}H gradient shimming, the sample stops spinning (gmapshim sets spin = 0).* ▪ *^{2}H gradient shimming converges after a few iterations. At each iteration, its # and r.m.s. error are informed in the text window.* ▪ *At each iteration, two ^{2}H profiles are displayed in the graphics window.* ▪ *The overall process takes a few minutes to complete* ▪ *^{2}H gradient shimming shims* ***Z1 to Z4*** *only. Any Z shim higher than Z4 and non-spin shims (X, Y, XZ, etc) are not changed.*	

5)	***Acquiring***	✓ For PROTON, click on *Setup → H1,CDCl3* (or *Setup→ H1→ Solvent*) and type ***su*** <ENTER> ▪ *For **CARBON**, click on Setup→ C13,CDCl3 (or Setup → C13→ Solvent) and type **su** <ENTER>* ✓ Type ***go*** <ENTER> to start acquisition. The sample spins at 20-25 Hz. ▪ *Number of scans (nt) default for PROTON is 16 and for CARBON is 1024. To increase the S/N of the spectrum by a factor of 2, increase nt by a factor of 4 **before** acquisition starts.* ▪ *If S/N is low, type nt = 1e6 <ENTER> **before** acquisition starts, and monitor the spectrum at every 4-scan block by typing **wft** <ENTER>.*	
6)	***Processing***	✓ Type ***wft*** <ENTER>	
7)	***Phasing*** ***(Automatic)***	✓ Type ***aph*** <ENTER> ▪ *Alternatively, type **aph0** <ENTER> to adjust the zero order phase parameter only.* *See step **12: Printing***	*Manual Phasing* ✓ Click on *Phase* button (below the *Main Menu* button) ✓ Using mouse's left-button, click once at the rightmost side of the spectrum. ✓ Holding the left-button, phase peaks up and symmetrically in this boxed region. ✓ Finish first step clicking on *Phase* button ✓ Resume first-order phasing clicking once at the rightmost side of the spectrum. Then click once at the leftmost side of the spectrum. Using the right button only, phase peaks up and symmetrically in this boxed region. ✓ Finish second step clicking on *Phase* button.
8)	***Peak picking***	✓ Click on *Th* button and adjust its vertical position to pick peaks of interest. ▪ *Press and hold the left button of the mouse and point it to the Th horizontal yellow line. Adjust it up or down accordingly.* ▪ *You may also type **th =** value <ENTER> at the command line, where value is the vertical position in mm.* ▪ *Peaks can be displayed by typing **dpf** <ENTER> .* ▪ *Sensitivity can be increased with **dpf(2)** or **dpf(1)**. Default is 3. The sensitivity may be increased, if a shoulder could not be picked with **dpf** only.* *See step **12a: Printing***	
9)	***Integrating***	✓ Click on *Partial Integral → Reset*. ▪ *A dotted green integral curve will appear on the spectrum.* ✓ Integrate each peak by placing the red vertical cursor red downfield to the peak (to the left) and, then, upfield to it (to the right). Repeat procedure for all peaks. ✓ After integration is completed, type ***bc*** <ENTER> for baseline correction. ▪ *To clear all resets points, type **cz** <ENTER>* *See step **12a: Printing***	

10)	***Saving FIDs***	✓ Type ***svf*** <ENTER> and, at the question mark, type in the name of the spectrum. ▪ *VNMR will append **.fid** as a suffix to the filename.* ▪ *VNMR saves each spectrum as a directory, composed of 4 files: 1) **fid:** the raw FID data, 2) **procpar:** contains all acquisition, processing, display parameters, 3) **log:** text file recording the history of the acquisition, and 4) **text:** text file containing the text name associated with the spectrum* [input via text('…') or gettext window, see Table on page 4].
11)	***Exiting VNMR program and UNIX Session***	✓ Eject your sample from the magnet, and put back in the standard sample ✓ *Lock* on the standard sample ✓ Type ***exit*** <ENTER> at the VNMR command line and answer the questions that appear in the confirmation windows ✓ After the VNMR program is exited, click with the RIGHT button of the mouse anywhere at the BACKGROUND: in the window of options, select **Exit** to finalize your **UNIX** session (**)
(**) *It is important NOT to forget to also exit the UNIX session because your spectrometer time charge is based on the length on this session.*		
12)	***Printing***	✓ Type ***pl pscale ppa page*** *<ENTER>* ▪ ***pl** (plot spectrum), **pscale** (plot scale), **ppa** (plot parameters), **page** (clear buffer).* *The above commands send plot objects to a plot buffer.*
12a)	***Printing***	✓ *Other commands:* ***ppf** (plot peaks)* ***pir** (plot integrals (needs **vp** 𝑜 12)*
12b)	***Printing***	✓ *Anything written in the text window can be re-directed to the printer:* ▪ *For example, type **printon dg printoff** <ENTER>* • ***printon*** *(turn print buffer **on**)* ***dg*** *(**d**isplay main **g**roup of parameters)* ***printoff*** *(clear print buffer and send to printer)* *Other commands: **dg1** (display group 2 -second page main parameters), **dgs** (display group shims), **dll** (display line list)*

OBS:

1) For poor **lock signal** and/or **lineshape**, do the following:
   a. Load stored shim settings by typing ***callshim*** <ENTER> at the VNMR console line.
   b. Click on *Acqi* to shim the sample manually.

2) ***gmapshim*** <ENTER> retrieves a shim file called *stdshims* before ^{2}H gradient shimming.

Manual Referencing – Find the solvent or TMS peak; click on the peak, and type at the VNMR command line:

nl rl*(xx<p>)<ENTER>.*

Example: ***rl***(7.27p) *for* $CDCl_3$

Some Useful VNMR Commands and Parameters	
xx ?	*Parameters can be checked at the command line by typing the parameter name with a question mark at the end.* *They can be set by typing the parameter name followed by an equal sign followed by the value, e.g. vs? nt? fn?*
vs	*Vertical scaling: can be set via middle button of the mouse.* *Position the mouse on the peak and increase its vertical scale by holding the middle mouse button.* *Parameter can also be changed at the keyboard, i. e.* ***vs=300.***
vp	*Vertical position, in mm from bottom of page (or screen).*
vsadj	*Automatically adjusts vs to fit the screen with the tallest peak of the spectrum.*
dscale	*Show scale below spectrum on screen (****pscale*** *to plot).*
text('....') *or type* ***gettext*** *<ENTER>*	*Make comment for file archiving and plotting*
cd<('path')>	*Change directory without an argument it sets the default directory to your login directory*
pwd	*Print working directory, show the current default directory*
dir	*Directory listing in text window*
cexp(#)	*Create experiment job #, where # is a number between 1 and 9*
jexp#	*Join experiment job #.* *Obs: all experiment jobs but exp1 are deleted upon logout*