Quick Guide for New Users

created 12/01/97 – updated 9/27/99

I. Login

- Username: practice Password: *[do not use 9th floor password; nothing from a dictionary; at least one number; case sensitive]*
- data to → /zhadum/practice [other partitions: europa, ganymede, starbase]

  UNIX: **mkdir** ***name***
  VNMR: **FILE** ***left-click on name*** **CHANGE** (will place data in ~practice/*name*)
- shims, macros, etc. to /export/home/practice/vnmrsys/shims or ~/vnmrsys/maclib, etc.
- exit VNMR before logging out!!

II. Setup

A. 1st Time:

- **MAIN MENU SETUP 1H,CDCL3** ; this will put reasonable parameters in
- **phasing=100** ; shows complete spectrum while phasing
- **MAIN MENU MORE CONFIG SELECT PLOTTER** **Shadowp_plot** or **Vorlonp_plot**
  SELECT PRINTER **Shadowp_print** or **Vorlonp_print**

B. Parameters:

- Setup probe and pulsed-field gradient parameters using macro with probe name:

  e.g., type **bbswg** produces **probe= 'bbswg' pfgon='nny'**
  bbold → probe='bbold' pfgon='nnn'
  hcx → probe='hcx' pfgon='nny'

  It is *critical* the probe parameter is set for correct parameters to be setup.

- **MAIN MENU SETUP Nuc,Solv**

  ^{1}H should be ok; check **nt** and **gain**

  ^{13}C – check decoupler settings, e.g., **probe='bbswg'** (make sure this is the probe installed)
  dmm='w'
  dmf=10000
  dpwr=40
  dm='yyy' su turns decoupler on (see also **UWMACROS DECOUPLER ON**)

C. Variable Temperature

- switch to N_2 gas for 20°C > temp > 40°C
- ***turn VT flow up to eject samples*** (do not switch back to air unless close to ambient)

- Use **UWMACROS SET TEMP** to change temps (or macro **settemp** or similar)

  UWMACROS SET TEMP should be used instead of manually setting temperature; this macro avoids inadvertant temperature changes that can otherwise occur.

- ±20° changes take 15 mins or so before probe tuning and shims will be stable.
  ±50° changes take ~30 mins.
  ±100° changes may take 1 h (should be done in steps no bigger than ±50°).
  - I***t is the student's responsibility to finish early enough that their VT work does not affect the next user!!***

- ***hcx probe -80 to +60°C***
  bbswg probe -150°C to +80°C
  bbold probe -150 to +150°C
  h1f19 probe -150 to +150°C

III. Locking and Shimming

A. Sample Prep:

- Need ≥ 0.6ml (4 cm height) solvent for Varian probes to attain good ^1H shims (without extraordinary shimming).
- Set sample to **67 mm** below bottom of spinner (use ruler), or
  center in rf coil region (use Varian depth gauge) if solvent < ~5 cm high.
- On a 500, it is critical that the sample be clear (no particulate floating if possible), and the tube be of high quality (Wilmad 506 minimum, 528 better) with no nicks or scratches. Keeping within these standards will allow excellent quality shims to be attained in ≤ 5 min shimming. Reasonable shims can be attained in other conditions, but with longer shimming sessions, and no guarantee that good quality lineshapes can ever be achieved.

B. Locking:

- use **UWMACROS LOADSHIMS** or manually use **rts**↵ or

- **rts('hcx.shim') loadshims** (loadshims → load='y' su load='n' su)
  rts('bbswg.shim') loadshims
  rts('*your-shims*') loadshims
  [**FILES SAVE SHIMS** or **svs**↵ will save in /export/home/practice/vnmrsys/shims]

- in ACQI window: set **FIELD** → until fid on-resonance (no oscillations) and positive
  LOCK POWER (see START suggestion, but be aggressively higher if needed)
  LOCK GAIN

  NOTE: IF ACQI WINDOW DOES NOT APPEAR, USE 'ACQI' TO CALL IT UP.

- should lock up now
- turn **SPIN ON** if routine 1D (<u>not</u> if 2D or selective 1D experiments)

C. Shimming:

- shim: **LOCK PHASE** ® **Z1** ® **Z2** to achieve maximum lock signal
- lower LOCK POWER (approaching FINAL suggestion; but keep lock level > 15)

- spinning: **LOCK PHASE** ® **Z1** ® **Z2** (2^{nd} order) ® **Z3** (usually not necessary)
  non-spinning **X** ® **Y** ® **XZ** (2^{nd} order) ® **YZ** ® **XY** ® **X2-Y2** , then back to above (2D do all without spinning)

- check shims using standard ^{1}H setup with **nt=1**
  set cursor on solvent singlet, **nl dres** ; should be ≤ 1 Hz in most cases; highly dependent on tube; 507's (stockroom) will sometimes need Z3+Z4 adjustments; 528's typically should not)

IV. Probe Tuning and ^{1}H Calibration

- Use Hewlett-Packard scope to tune probes:
  - disconnect ^{1}H cable (with small silver barrel filter attached) from probe, and hook HP scope into probe
  - push H1 PROBE on the scope
  - tune in TUNE capacitor (bottom) to center dip

    tune in MATCH capacitor (upper) to get dip down to bottom 2-3 squares

    readjust both to center and bottom dip appropriately
  - disconnect scope cable and reconnect ^{1}H
  - tune ^{13}C or other X nucleus if needed at this time
- check ^{1}H pulsewidth using **array** command: use **pw 30 3 3** to set up first array; check about 360° (going negative to postive as **pw** increases); **pw90 = pw(@ 360°) / 4**
- pw90 check is required for checkout; also necessary before querying facility staff about probe

V. Acquisition

- Check that there is no external attenuation in-line for ^{13}C or other nuclei runs.
- Set **gain=40** and listen/watch for an ADC OVERFLOW beep [there is one beep for completion of the acquisition, and a second beep if there is an ADC overflow]; turn gain down in 10 dB steps until ok (this is recommended setup; computer sees clipping better than you will in next example!).
- Can also perform a **nt=1** acquisition, then **df**; if fid looks normal, type **gf** (wait > 2s!!!), then can go into **ACQI FID** and observe fid directly while changing gain.
- **go** or
  ga (will automatically wft) or
  au (will perform additional commands useful for 2D or auto-saving data)

- ^{1}H acquisition usually needs only a check of **nt**.
  - **movesw** similar to **^O** in EP on AM/AC's
  - **movetof** similar to changing **O1** on AM/AC's

- ^{13}C, turn on decoupler: **dm='yyy' su**
  - remember to turn off when done!
  - set **nt=1e6** if don't know needed number of scans
    can **wft** after each **bs** scans (**lb=2-4** needed for ^{13}C)
    use **sa** to stop acquisition once S/N is good enough
- cursor close to peak, **nl rl(77p)** will correctly reference $CDCl_3$ peak; **rl1(77p)** for *f1* in 2d's
- **dsx** → wft dscale(-3)
- **ppa pscale pl page** typical plot

VI. Saving and Deleting Data, and Backups

- **FILES SAVE FID** or **svf('*data-name*')** ....; saves only raw fid but with *all* parameter's intact
- **MAIN MENU FILE SAVE FID** type in name without quotes
- **MAIN MENU FILE *left-click on data-name* DELETE** will delete data
- use FTP program on PC's and connect to ZIP's to backup

VII. Logging Out

- exit VNMR first
- *right-click* on background, exit

VIII. Don'ts

- use Unity without VT regulation (run at 26°C if just want ambient)
- use too little solvent; Varian probes require more solvent than Bruker probes; too little solvent will just give you a terrible shimming session
- run VT outside 20 to 40°C without switching over to N_2 gas
- run VT below 10°C without Variac on
- run VT outside -80 to +60°C on hcx probe, -120 to +80°C on bbswg probe