Operation of Unity–500

created 11/7/95 – updated 10/16/98

I. Proper Exiting and Logging In and Initial VNMR Setup

- if an experiment is running, and your time is clearly in effect, use:
  svf('*savename*') while experiment is running–will save last **bs** dataset
  use FileManager to check that file wrote correctly (unix command **df** better)
- **exit** vnmrx, right-click-and-hold on background and release on EXIT to exit CDE
- do *not* save the workspace while VNMRX is open

II. Commands for First Time and Novice Users

Some of the following commands/procedures may have to be performed when first starting; many of them will not be needed again (or only occasionally):

- **phasing=100** (or =60 on a Sparc1 if the data is >64k)
- **cexp(2) cexp(3) ...** create additional experiment areas (see also MAIN WORKSPACE CREATE)
- click MAIN MENU MORE CONFIG PRINTER and keep clicking PRINTER until set to Shadowp_LJ (laserjet portrait printing)
  – repeat above except for PLOTTER and click unter set to Shadowp_LJR (landscape plotting)
- **gf** following correct setup to give good fid/spectrum shimming inside ACQI FID window

III. Probe Changes

ONLY FOR TA'S AND FEW STUDENTS OK'ED FOR PROBE CHANGES

- see Table 1 on the next page for a description of probes
- make sure acquisition is complete and data saved by previous user
- stop temperature control by using macro **tempoff** (in /vnmr/maclib)
- physically switch the temp controller off
- eject sample (type **eject** at command prompt, or click **eject** inside shimming/acqi window); type **insert** to turn the air back off
- disconnect rf cabling, VT line, and probe cooling tygon
- disconnect temp/heating cable using blue nonmagnetic screwdriver
- unscrew two probe thumbscrews and guide probe out
- insert correct probe; use care with last 1" — you may have reseat aluminum bore tube by pushing gently downward pressure at top of magnet (necessary if sample won't spin)
- reconnect cables: *keep Nalorac cable and filters separate and use only for that probe*
- power up and restart temp control with UWMACROS SET TEMP or macro similar to **temp24**
- read in new shims and load, e.g.:
  rts('triple') loadshims (better use **UWMACROS LOADSHIMS**)
- change probe and pfg settings appropriate for probe:
  probe='hcx' pfgon='nny'
  probe='bbold' pfgon='nnn'
  probe='1h19f' pfgon='nnn'
  probe='3mm' pfgon='nny' (UNITY) **pfgon='yyy'** (INOVA)

Table 1. Description of Probes on Unity-500 and Inova-500

Use the following general rules for probe selection:

- concentration limited samples: use the largest diameter probe appropriate to the experiment
  - hcx or $^{1}H/^{19}F$ best for 1d or homonuclear ^{1}H experiments
- quantity limited samples: use the smallest diameter probe appropriate to the experiment
  - Nalorac 3mm probe best for all ^{1}H experiments in this case
  - ***strongly* consider using susceptibility-matched inserts for 3mm ^{1}H or 5mm X experiments (~3´ saving in amount of material needed to obtain a particular S/N in a fixed amount of time, or ~10´ decrease in time for fixed amount of material!!)**

Name	Type	Temp	Description
hcx	5mm 1H {13C, X}	≥ -80°C ≤ +60°C	• inverse triple with PFG • good for ^{1}H and ^{1}H-X heterocorrelation • excellent 1H S/N~800
bbold	5mm broadband	≥ -150°C ≤ 150°C	• standard probe for direct ^{13}C, ^{31}P, ^{29}Si, ... observation • ^{1}H signal-to-noise (S/N) and line shape are poor with this probe
bbswg	5mm broadband switchable (i.e. 1H observe) with pfg	≥ -130°C ≤ +60°C	• standard probe for direct ^{13}C, ^{31}P, ^{29}Si, ... observation • ^{1}H S/N is adequate with this probe, so probe switching for ^{1}H observation is not needed (for best ^{1}H S/N, use inverse or h1f19 probe; bbswg 1H S/N~350)
h1f19	5mm $^{1}H/^{19}F$	≥ -150°C ≤ 150°C	• for best sensitivity ^{1}H work when concentration is limited • ^{1}H S/N is good (540 on EB)
nal3mm	3mm nalorac (INOVA only)	≥ -40°C ≤ 40°C	• for ^{1}H 1D and 2D heterocorrelation ($^{13}C/^{15}N$ only) when sample amount is limited, or need best water suppression • ^{1}H S/N is very good (5mm probes are better for concentration limited samples)
invx	5mm inverse broadband	≥ -150°C ≤ 150°C	• for 2D hetercorrelation work: HMQC, HMBC, HSQC • ^{1}H signal-to-noise is very good with this probe • X S/N is poor with this probe; do not do ^{13}C observe with this probe
triple	5mm triple	≥ -100°C ≤ 100°C	• for 2D ^{1}H–^{13}C–^{15}N work: HMQC, HMBC, HSQC • ^{1}H S/N is very good • ^{13}C and ^{15}N S/N is poor • VT range is limited: -50 to +80°C

Table 2. Calibrations of Probes on Unity-500

Use the following guidelines for probe calibrations:

- short runs use facility numbers (see /vnmr/shims/probes* file for up-to-date numbers)
- ^1H pw90 checks are always recommended time permitting for all experiments
- if probe problems are suspected, check pw90's of X and ^1H observe (not decouple)
- always perform calibrations (at minimum ^1H pw90 check) for overnight or longer runs for PT-type experiments;
- for standard decoupling, calibrations are rarely needed even for long runs (although having pw ~< 90° is best)

See file on-line	/narn/vnmr5.3b/shims/probes*.....if not logged onto narn /export/home/vnmr/shims/probes* .....if logged onto narn

- (preferable to the following is UWMACROS LOADSHIMS or FILES DATA SHOWSHIMS)

  shim files that are available can be listed by entering the following commands in a UNIX window:

  - facility shim files: type **dir /vnmr/shims**
  - your shim files: type **dir ~/vnmrsys/shims**
  - *user* shim files: type **dir /home/*user*/vnmrsys/shims**

  or simply go to path in the FileManager
- load shims with UWMACROS LOADSHIMS

III. Probe Tuning

- recommended method: **UWMACROS TUNE PROBE ...**

or enter macro similar to **tuneh** (in /vnmr/maclib)

- **gain=0** is necessary for tuning (UWmacros restore original gain setting)
- make sure decoupler is off (**dm='n' su** if necessary)
- move cable (e.g., ^1H) from **obs** or **dec** BNC to **tune** BNC
- switch knob from **obs** to **tune**
- adjust tune and match to achieve 0 on meter (in most cases, getting needle < 10 is sufficient)

On many of the probes, there will be three capacitors:

It is essential that the two similar capacitors stay at nearly the same capacitance (i.e., same number of turns from end), so make sure to move them together

For example, on the bbold probe, the ^1H channel has a gold and two silver rods (all small diameter) connected to capacitors. The silver are both "match" capacitors, and must therefore be turned together: if you move one clockwise by ¼ turn, the other should also be turned clkwise ¼ turn.

- switch knob back to **obs**
- move cable back to **obs** or **dec** BNC
- tune other channels as needed

inverse ^1H/X probe: for ^1H channel, tune the gold (match) and silver (tune1) knobs first, then make sure black knob (tune2) is within ¼ turn of silver knob

IV. Lock and shim

Use care when clicking on* CONNECT *on the acquisition window; fast clicking can crash the computer (requiring up to 30 min to correctly reboot!), so use patience when going to acqi

- click into the LOCK panel in the acqi window and turn off the lock
- change Z0 until there is no oscillation in the lock signal: do not hesitate to turn up lock power and lock gain achieve lock, but lower LOCK POWER as soon as possible to avoid lock saturation
- set the LOCK POWER to recommended settings (only go up to potentially safe setting if shims are poor; set back once shims have improved) and use LOCK GAIN thereafter to adjust amplitude
- adjust LOCK PHASE analogous to a shim to get positive going signal
- turn on LOCK
- adjust LOCK PHASE as a shim to maximize lock signal (make sure to return to LOCK PHASE fairly often when shimming, especially after large changes in Z2)
- click into SHIM window and shim normally
  - start by 1st order shimming Z and Z2; when finished take nt=1 acquisition to check line shapes use **nl dres** or if S/N is excellent use **nl res** to get indication of line shape
  - target 50% full linewidth ≤ 1 Hz for most samples, spinning or non-spinning
  - now 2nd order shim Z2 (choose a direction to move Z2; this will decrease lock signal [1st order shim had lock signal maximized at current Z2]; see if Z1 improves; if so continue, if not go other direction in Z2)
  - shim X Y XZ XY XY X2-Y2 all 1st order, then repeat 2nd order Z Z2 shim
  - check line shape; if not at target try spinning sample; if improves considerably turn spin off and work on X Y shims; if did not improve much with spinning then need to target higher order Z's
- Table 4 shows shims dependencies for the Unity-500; 2nd order shimming is required on all 500 MHz instruments (i.e., you simply cannot expect to get a good shim without it)

Table 3. Field and Lock Power Settings for Unity–500

solvent	^1H δ(ppm)	Z0 (field as of 97/12/01)	FINAL lock power	STARTING lock power
acetonitrile-d3	1.93(5)	630	10	20
acetone-d6	2.04(5)	500	12	20
dimethysulfoxide-d6	2.49(5)	50	14	24
deuterium oxide-d2	4.63(DSS)	2700	28	40
methylene chloride-d2	5.32(3)	-3400	15	25
benzene-d6	7.15(br)	-5600	18	30
chloroform-d3	7.24(1)	-5700	25	35

☞ Z0 will change by ~ +100 units each week.

Table 4. Major Shim Interactions on Unity–500

[+ means shim move in same direction—positive change in Z4 results in positive change in Z2]
Much of the table is not completed; since new shims installed, most interactions are now much weaker.

Adjusted shim	Stong interaction		Weak interaction		Adjusted shim	Stong interaction	Weak interaction
Z^5	Z^3	?	Z^4	?	XZ	X	Z
Z^4	Z^2	-	Z^3	?	YZ	Y	Z
Z^3	Z	?	Z^2	?	Z^2X	ZX	Z, X^3
Z^2	Z	-			Z^2Y	ZY	Z, Y^3, ZXY
					X^2Y^2	XY	

Table 5. Shim Sensitivities on Unity–500

[number following shim is normal adjustment when shim fairly close to correct]

Sensitive		Moderate				Insensitive	
Z	16 to 4	Z^3	64 to 16	XY	64	X^3	64
Z^2	16 to 4	Z^4	64	Z^2X	64	Y^3	64
Y	16 to 4	X	16	Z^2Y	64	ZX^2Y^2	64
YZ	16	XZ	16	ZXY	64		