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 vnmx`, 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 `/vnmx/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)
- Disconnect 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 \(^1H/^{19}F\) best for 1D or homonuclear \(^1H\) experiments
- quantity limited samples: use the smallest diameter probe appropriate to the experiment
  - Nalorac 3mm probe best for all \(^1H\) experiments in this case
  - strongly consider using susceptibility-matched inserts for 3mm \(^1H\) 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!!)

<table>
<thead>
<tr>
<th>Name</th>
<th>Type</th>
<th>Temp</th>
<th>Description</th>
</tr>
</thead>
</table>
| hcx    | 5mm 1H \{13C, X\}  | ≥ -80°C ≤ +60°C | • inverse triple with PFG  
• good for \(^1H\) and \(^1H\)-X heterocorrelation  
• excellent \(^1H\) S/N~800 |
| bbold  | 5mm broadband       | ≥ -150°C ≤ 150°C | • standard probe for direct \(^13C\), \(^31P\), \(^29Si\), ... observation  
• \(^1H\) 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 \(^13C\), \(^31P\), \(^29Si\), ... observation  
• \(^1H\) S/N is adequate with this probe, so probe switching for \(^1H\) observation is not needed (for best \(^1H\) S/N, use inverse or h1f19 probe; bbswg \(^1H\) S/N~350) |
| h1f19  | 5mm \(^1H/^{19}F\)  | ≥ -150°C ≤ 150°C | • for best sensitivity \(^1H\) work when concentration is limited  
• \(^1H\) S/N is good (540 on EB) |
| nal3mm | 3mm nalorac (INOVA only) | ≥ -40°C ≤ 40°C | • for \(^1H\) 1D and 2D heterocorrelation \((^{13}C/^{15}N\) only) when sample amount is limited, or need best water suppression  
• \(^1H\) S/N is very good (5mm probes are better for concentration limited samples) |
| invx   | 5mm inverse broadband | ≥ -150°C ≤ 150°C | • for 2D heterocorrelation work: HMQC, HMBC, HSQC  
• \(^1H\) signal-to-noise is very good with this probe  
• X S/N is poor with this probe; do not do \(^13C\) observe with this probe |
| triple | 5mm triple          | ≥ -100°C ≤ 100°C | • for 2D \(^1H-^{13}C-^{15}N\) work: HMQC, HMBC, HSQC  
• \(^1H\) S/N is very good  
• \(^13C\) and \(^15N\) 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 $< 90^\circ$ is best)

See file on-line

<table>
<thead>
<tr>
<th>Facility Shim Files</th>
<th>Type Command</th>
</tr>
</thead>
<tbody>
<tr>
<td>/vnmr/shims</td>
<td>dir /vnmr/shims</td>
</tr>
<tr>
<td>~/vnmrsys/shims</td>
<td>dir ~/vnmrsys/shims</td>
</tr>
<tr>
<td>/home/username/vnmrsys/shims</td>
<td>dir /home/username/vnmrsys/shims</td>
</tr>
</tbody>
</table>

- (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/username/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 $\frac{1}{4}$ turn, the other should also be turned clockwise $\frac{1}{4}$ 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 $\frac{1}{4}$ 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 acquisition 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 to 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 n=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 \( \leq 1 \) 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

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

\( ^{1}H \) 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.

<table>
<thead>
<tr>
<th>Adjusted shim</th>
<th>Strong interaction</th>
<th>Weak interaction</th>
<th>Adjusted shim</th>
<th>Strong interaction</th>
<th>Weak interaction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Z^3</td>
<td>Z^3 ?</td>
<td>Z^4 ?</td>
<td>XZ</td>
<td>X</td>
<td>Z</td>
</tr>
<tr>
<td>Z^4</td>
<td>Z^2 -</td>
<td>Z^3 ?</td>
<td>YZ</td>
<td>Y</td>
<td>Z</td>
</tr>
<tr>
<td>Z^3</td>
<td>Z ?</td>
<td>Z^2 ?</td>
<td>Z^2X</td>
<td>ZX</td>
<td>Z, X^3</td>
</tr>
<tr>
<td>Z^2</td>
<td>Z -</td>
<td>Z^2Y</td>
<td>ZY</td>
<td>Z, Y^3, ZXY</td>
<td></td>
</tr>
</tbody>
</table>

Table 5. Shim Sensitivities on Unity–500

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

<table>
<thead>
<tr>
<th>Sensitive</th>
<th>Moderate</th>
<th>Insensitive</th>
</tr>
</thead>
<tbody>
<tr>
<td>Z 16 to 4</td>
<td>Z^3 64 to 16</td>
<td>X^3 64</td>
</tr>
<tr>
<td>Z^2 16 to 4</td>
<td>Z^4 64</td>
<td>Y^3 64</td>
</tr>
<tr>
<td>Y 16 to 4</td>
<td>X 16</td>
<td>ZX^2Y^2 64</td>
</tr>
<tr>
<td>YZ 16</td>
<td>XZ 16</td>
<td>ZXY 64</td>
</tr>
</tbody>
</table>