1D NMR Acquisition Quick Guide

1. Getting Started:
   - Logon using your Username and Password.
   - Click on the VNMR Icon ( ).
   - Type \texttt{e <rtn>}. Place sample in spinner.
     Gauge properly. Place on top of magnet. Type \texttt{i <rtn>}
   - Click Setup=>\textbf{H1,CDC13} (or desired expt.).
   - Type \texttt{fixshims <rtn>}. Wait for beep.
   - Click \textbf{acqi}. A new window will pop up.
   - In new window, click \textbf{LOCK}.
   - Check spinning. It should be 20.

2. Establishing Lock and Shimming:
   - Click \textbf{LOCK off}.
   - Move \textbf{lockpower} and \textbf{gain} slider to maximum.
   - Move \textbf{Z0} slider slowly until one ‘beat’ is visible.
   - Click \textbf{LOCK on}.
   - Adjust \textbf{lockphase} to maximize lock level.
   - Click \textbf{SHIM}.
   - Adjust \textbf{Z1C} -1+ to maximize lock level.
     Repeat on \textbf{Z2C} -1+. Continue to alternate until no improvement. (Skip if no \textbf{Z1C}, \textbf{Z2C}).

3. Shimming (continued):
   - Adjust \textbf{Z1} -64+ until maximum. Repeat with \textbf{Z2}.
   - Adjust \textbf{Z1} -16+ until maximum. Repeat with \textbf{Z2}.
   - Adjust \textbf{Z1} -4+ until maximum. Repeat with \textbf{Z2}.
   - Click \textbf{CLOSE} to exit the lock/shimming window.
   - Type \texttt{nt=1 ga <rtn>}. (Use with \textit{1H} spectra only.)
   - When complete, type \texttt{f full aph <rtn>}, expand around solvent peak or suitable well-resolved singlet. Type \texttt{vsadj <rtn>}
   - Is this peak well shimmned (i.e. is it narrow, symmetric, etc.)? If yes, proceed to acquisition. If not, click \textbf{acqi} then \textbf{SHIM}.
   - Adjust appropriate shim (e.g. \textbf{Z1} for symmetric broadening or \textbf{Z2} for asymmetric peak shape).
   - Repeat single scan acquisition. Reshim, if necessary.

4. Acquiring Your Spectrum:
   - Type \texttt{nt=32} (or desired scans in multiples of 4) <rtn>. If you want accurate integration, increase default \texttt{d1} (e.g. \texttt{d1=10}).
   - For \textit{13C}, use \texttt{nt=1e6 bs=8}.
   - Type \texttt{ga <rtn>}.
   - For \textit{13C}, after a few data blocks are complete (message: BS # completed), type \texttt{wft} to process. When sufficient S/N is obtained, stop with \texttt{sa(‘bs’)}.
   - When complete, type \texttt{f full aph vsadj <rtn>}

5. Referencing Your Spectrum:
   - Type \texttt{dscale <rtn>} and find your solvent peak (use the reference chart below or one near the instrument).
   - With \textbf{left} mouse button click to the \textbf{left} of solvent peak.
   - With \textbf{right} mouse button click to the \textbf{right} of solvent peak.
   - Click \textbf{Expand}.
   - Place cursor on top of solvent peak.
   - Type \texttt{nl rl(‘<your solvent ppm>’p) <rtn>}. For example, for CDC13 you would type \texttt{nl rl(7.24p) <rtn>}

Common Deuterated Solvents:

<table>
<thead>
<tr>
<th>Solvent</th>
<th>\textbf{ppm}</th>
<th>\textbf{ppm}</th>
</tr>
</thead>
<tbody>
<tr>
<td>\textbf{CDCl}_3</td>
<td>7.24p(\textit{H})</td>
<td>77p(\textit{13C})</td>
</tr>
<tr>
<td>\textbf{Acetone-d}_5</td>
<td>2.04p(\textit{H})</td>
<td>29.8p(\textit{13C})</td>
</tr>
<tr>
<td>\textbf{Benzene-d}_6</td>
<td>7.15p(\textit{H})</td>
<td>128.0p(\textit{13C})</td>
</tr>
<tr>
<td>\textbf{DMSO-d}_6</td>
<td>2.49p(\textit{H})</td>
<td>39.5p(\textit{13C})</td>
</tr>
<tr>
<td>\textbf{CD}_3CN</td>
<td>1.93p(\textit{H})</td>
<td>1.3p(\textit{13C})</td>
</tr>
<tr>
<td>\textbf{D}_2O</td>
<td>4.63p(\textit{H})</td>
<td>\textbf{ppm}</td>
</tr>
<tr>
<td>\textbf{CD}_3OD</td>
<td>3.30p(\textit{H})</td>
<td>49.0p(\textit{13C})</td>
</tr>
<tr>
<td>\textbf{CD}_2Cl_2</td>
<td>5.32p(\textit{H})</td>
<td>53.8p(\textit{13C})</td>
</tr>
<tr>
<td>\textbf{DMF-d}_7</td>
<td>2.91p(\textit{H})</td>
<td>35.2p(\textit{13C})</td>
</tr>
<tr>
<td>\textbf{CD}_3CN</td>
<td>2.74p(\textit{H})</td>
<td>30.1p(\textit{13C})</td>
</tr>
</tbody>
</table>
Integrating Your Spectrum (Not for 13C NMR):
• Type *f full* <rtn>. Click Part Integral or Part Int.
• Type *cdc dc cz* <rtn>.
• Expand around first desired integral region.
• Click Resets.
• Use a left mouse click for each integral reset point. If you make a mistake, use the right mouse button to undo last reset point. To restart, type *cz* <rtn>.
• Click Full, expand the next integral region, click Resets, left click your reset points, repeat for every region.
• When complete, click Full.

Referencing Your Integrals:
• Expand around Integral to be referenced.
• Place cursor on an integral region. The red vertical cursor must be on an integral trace.
• Click Set Int and enter an integral value (e.g. 2.0 <rtn>).
• Type *vp=15* <rtn>.
• Type *ds f full dir* <rtn> to display your integrals.
• Type *ds* <rtn> to get an interactive spectrum.

Saving Your Data:
• Type *svf('your filename')* <rtn>.

Peak Picking:
• Type *f full ds* <rtn>. Click Th and place yellow threshold line below top of smallest desired peak.
• Type *dpf* <rtn>. If too many peaks, type *ds* <rtn>, click Th and move threshold up. Type *dpf* <rtn> to recheck.

Add Text to Your Spectrum:
• Type *text('your text here\: more text on new line\: even more text on a third line')* <rtn>.

Logging Off of a Session:
• Type *e* to eject sample.
• Place standard in spinner. Gauge properly. Place on top of magnet.
• Type *l* <rtn>.
• Type *exit* <rtn>.
• Click the EXIT button on screen bottom, click OK.

Analyze Your Spectrum:

Common Plotting Commands:
- *pl* plot spectrum
- *pscale* plot scale
- *pir* plot integral regions
- *ppf* plot peak frequencies
- *pll* plot line list with freqs in Hertz
- *pltext* plot text
- *pltext(150,150)* plot text in top right (use with *pll*)
- *pap* plot all parameters
- *page* send plot to printer
# VNMR Basic Commands

<table>
<thead>
<tr>
<th>Command</th>
<th>Description</th>
<th>Typed Example</th>
</tr>
</thead>
<tbody>
<tr>
<td>nt</td>
<td><strong>number of transients</strong>: Sets the number of transients (scans) to be acquired. You should always select a multiple of 4 (e.g. 4, 8, 128). The larger the number of scans, the better the signal to noise.</td>
<td>$nt=16$ : default setting for 1H,CDCl3</td>
</tr>
<tr>
<td>bs</td>
<td><strong>block size</strong>: Directs the acquisition computer, as data are acquired, to periodically store a block of data on the disk.</td>
<td>$bs=8$ : sets the block size to 8 scans.</td>
</tr>
<tr>
<td>ga</td>
<td><strong>submit experiment to acquisition and FT the result</strong>: Performs the experiment described by the current acquisition parameters and Fourier transforms (<code>wft</code>) the result.</td>
<td>$ga$</td>
</tr>
<tr>
<td>wft</td>
<td><strong>weight and Fourier transform 1D data</strong>: Performs a Fourier transform on one or more 1D FIDs with weighting applied to the FID.</td>
<td>$wft$ : used if you stop the acquisition prior to completion or when loading a saved FID.</td>
</tr>
<tr>
<td>aph</td>
<td><strong>automatic phase of rp and lp</strong>: Automatically calculates the phase parameters $lp$ and $rp$ required to produce an absorption mode spectrum and applies them to the current spectrum.</td>
<td>$aph$ usually gives well phased spectra</td>
</tr>
<tr>
<td>f, full</td>
<td><strong>full</strong>: Sets the horizontal and vertical control parameters to produce a display on the entire screen.</td>
<td>$f$ or $full$</td>
</tr>
<tr>
<td>vsadj</td>
<td><strong>Automatic vertical adjustment</strong>: Automatically sets the vertical scale, $vs$, in the absolute intensity mode so that the largest peak is at the requested height.</td>
<td>Vsadj : resets the vertical scale to fit on the screen</td>
</tr>
<tr>
<td>dscale</td>
<td><strong>Display scale below spectrum or FID.</strong></td>
<td>$dscale$</td>
</tr>
<tr>
<td>aa</td>
<td><strong>abort acquisition</strong>: immediately aborts the acquisition.</td>
<td>$aa$</td>
</tr>
<tr>
<td>sa</td>
<td><strong>stop acquisition</strong>: stops acquisition after acquiring current transient.</td>
<td>$sa$</td>
</tr>
<tr>
<td>su</td>
<td><strong>submit a setup experiment to acquisition</strong>: Sets up the system hardware to match the current parameters but does not initiate data acquisition.</td>
<td>$su$</td>
</tr>
<tr>
<td>svf</td>
<td><strong>Save FIDs in current experiment</strong>: Saves parameters, text, and FID data in the current experiment to a file.</td>
<td>svf('H1_070703') : saves the FID as a file named H1_070703</td>
</tr>
</tbody>
</table>