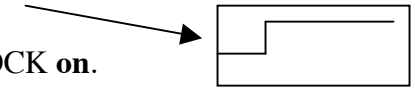
1D NMR Acquisition Quick Guide

1 Getting Started:

- Logon using your Username and Password.
- Click on the VNMR Icon( ).
- Type *e* <rtn>. Place sample in spinner. Gauge properly. Place on top of magnet. Type *i* <rtn>.
- Click **Setup=>H1,CDCl3** (*or desired expt.*).
- Type *fixshims* <rtn>. Wait for beep.
- Click **acqi**. A new window will pop up.
- In new window, click **LOCK**.
- Check spinning. It should be 20.

2 Establishing Lock and Shimming:

- Click LOCK **off**.
- Move **lockpower** and **gain** slider to maximum.
- Move **Z0** slider slowly until one 'beat' is visible.



- Click LOCK **on**.
- Reduce **lockpower** in units of 4 until correct value: 5-10 for D_2O, acetone-d_6; 10-15 for C_6D_6; 20-32 for $CDCl_3$.
- Adjust **lockphase** to maximize lock level.
- Click **SHIM**.
- Adjust **Z1C** -1+ to maximize lock level. Repeat on **Z2C** -1+. Continue to alternate until no improvement . (Skip if no **Z1C**, **Z2C**).

3 Shimming (*continued*):

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

4 Acquiring Your Spectrum:

- Type *nt=32* (or desired scans in multiples of 4) <rtn>. For ^{13}C, use *nt=1e6 bs=8*.
- Type *ga* <rtn>. For ^{13}C, after a few data blocks are complete (message: BS # completed), type *wft* to process. When sufficient S/N is obtained, stop with *sa('bs')*.
- When complete, type *f full aph vsadj* <rtn>.

Manual Phasing (*Optional*):

- If autophasing did not work, type *lp=0 rp=0* <rtn>.
- Click on **Phasing**. Using the ***left*** mouse button, click and hold on the ***Rightmost*** peak. Drag the mouse up or down to phase that peak.
- Using the ***right*** mouse button, click and hold on the ***Leftmost*** peak. Drag the mouse up or down to phase.
- Click **box**. If phasing is still poor, repeat above process.

5 Referencing Your Spectrum:

- Type *dscale* <rtn> and find your solvent peak (use the reference chart below or one near the instrument).
- With ***left*** mouse button click to the ***left*** of solvent peak.
- With ***right*** mouse button click to the ***right*** of solvent peak.
- Click **Expand**.
- Place cursor on top of solvent peak.
- Type *nl rl(<your solvent ppm>p)* <rtn>. For example, for $CDCl_3$ you would *type nl rl(7.24p) <rtn>*.

Common Deuterated Solvents:

$CDCl_3$	**7.24p(^1H) 77p(^{13}C)**	**D_2O**	**4.63p(^1H)**
Acetone-d_6	**2.04p(^1H) 29.8p(^{13}C)**	**CD_3OD**	**3.30p(^1H) 49.0p(^{13}C)**
Benzene-d_6	**7.15p(^1H) 128.0p(^{13}C)**	**CD_2Cl_2**	**5.32p(^1H) 53.8p(^{13}C)**
DMSO-d_6	**2.49p(^1H) 39.5p(^{13}C)**	**DMF-d_7**	**2.91p(^1H) 35.2p(^{13}C)**
CD_3CN	**1.93p(^1H) 1.3p(^{13}C)**		**2.74p(^1H) 30.1p(^{13}C)**

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6 Integrating Your Spectrum (Not for ^{13}C 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**.

7 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 *f full dpir* <rtn> to display your integrals.
- Type *ds* <rtn> to get an interactive spectrum.

Saving Your Data:

- Type *svf('your filename')* <rtn>.

8 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>.

9 Plotting Your Spectrum:

- Typical example, *pl pscale ppf pir pltext page* <rtn>.
- Type *ds* <rtn>, expand desired plot regions, and repeat plot command.

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

10 Logging Off of a Session:

- Type *e* to eject sample.
- Place standard in spinner. Gauge properly. Place on top of magnet.
- Type *i* <rtn>.
- Type *exit* <rtn>.
- Click the **EXIT** button on screen bottom, click **OK**.

11 Analyze Your Spectrum: