Instructions for the 500 MHz NMR Spectrometer

I. Sample Preparation

1. wipe exterior of tube with Kimwipe
2. choose an empty sample holder, pull or push inner barrel all the way down
3. insert tube into inner barrel
4. check height with height gauge
   - if tube hops in height gauge, twist tube or inner barrel to minimize hopping
5. place sample w/holder in an open slot in auto-sample tray
   - remember slot number

II. Initial Setup

1. log on to computer
2. open **Delta**
3. click **Spectrometer Control** icon 
4. in Spectrometer Control window, click **Sample**
5. in Sample window, change **Slot** number to desired slot
6. choose solvent in **Solvent** field

III. Locking - Manual

1. in Spectrometer Control window, click **Sawth** wait for lock signal to appear
   - Goal is to get vertical line of cross-hair lined up with largest (1st) peak of lock signal oscillation. To do this, either:
     A. click left/right arrows of **Shim Z0** to move cross-hair left/right, or
     B. in **Pick** side-menu, choose **Pick position** icon 
        - click and drag center cross-hair to desired position
   - click **Cancel** to finish
2. in Sample window, click **Lock on** icon 

IV. Shimming - Manual

1. near bottom of Sample window, go to **Shim_Z1**, click **+50x** or **-50x**
   - click one, observe lock level (green bar)
     • if increased, continue with that button
     • if decreased, switch to other button
   - continue until lock level is at its *maximum*
   - use fine control of **+10x** or **-10x** if necessary
2. repeat with **Shim_Z2**
3. readjust **Shim_Z1** if necessary

Auto-Lock and Auto-Shim Options

1. **Auto Lock** 
2. **Gradient Shim**  to automatically shim the sample
   - this infers your have previously locked the sample
3. **Auto Lock and Shim** 
4. **Gradient Shim and Lock**  to automatically lock and shim sample
   - gives best results of above options

** New users are encouraged to learn to *manually* lock and shim!

V. Acquisition

1. in Spectrometer Control window, click **Expmnt**
2. in new window, click folder with globe (global directory)
3. under **Filename** scroll down and click **single_pulse.ex2**, click green **Ok**
   - single_pulse.ex2 is for standard 1-dimensional ^{1}H acquisition
4. in Experiment Tool window, change/check the following

   Header tab
   - enter **filename**
   - enter **sample_id** and **comment** if desired
   - click **auto_gain**

   Instrument tab
   - check solvent

   Acquisition tab
   - change **x_points**, if desired
   - change **scans**, if desired

   Pulse tab
   - advanced users only
5. click green **Submit**
6. click **GO** in pop-up window
7. in Spectrometer Control window, click **View**

VI. Data Processing – Referencing the Spectrum

1. from the top menu: **Options → Show Options**
2. expand/zoom the reference peak
   - in **Zoom** side-menu, choose **Zoom data** icon 
   - in gray border area, click mouse, hold, scroll left → right, release
   - repeat zoom as necessary
3. in **X Reference:** field of **Options**, enter **0.00[ppm]** or other specific ppm reference
4. in **Reference** side-menu choose **Paste reference to nearest peak** icon 
5. click mouse near reference peak, done

VII. Data Processing – Peak Picking

1. zoom peaks of interest
2. in **Peak** side-menu, choose **Adjust peak threshold** icon 
3. click on spectrum, click-hold-and-drag green line up or down accordingly
   - peaks extending above the green line will be labeled
   - peaks below the green line will not be labeled
4. click  icon at top of window

VIII. Data Processing – Integration

1. in **Integral** side-menu, choose **Adjust baseplane/thresholds** icon 
2. click spectrum, click-hold-and-drag red line to desired height
   - red line indicates height at which integrals will be drawn
3. in **Integral** side-menu, choose **Create integral** icon 
   - work left → right along the green spectral baseline
   - to left of peak to be integrated, click-and-hold
   - drag cursor to right of peak to be integrated, release mouse
   - repeat for each peak of interest

   ** integrating the largest peak first in window usually gives best results
4. if mistake is made, in **Integral** side-menu, choose **Delete integral** icon 
   - click integral to be deleted, reintegrate
5. to reference an integral, in **Integral** side-menu, choose **Select integrals** icon 
   - click on integral to be referenced
   - in **Options**, enter value of integral in **Normal** field

IX. Printing – What You See Is What You Get!

1. expand peaks of interest
   - zoom, or
   - enter upper [ppm] bound in **X Start:**, enter lower [ppm] bound in **X Stop:**
2. click **Plot** icon  at top of window
3. click green **OK** in pop-up window
4. to turn off parameter plotting, **Preferences** → unclick **Plot Params**

X. Exit

1. in Sample window, change **Slot** number to **1**
   - ejects your sample, inserts D_2O standard
2. in **Solvent** field, choose **D_2O**
3. click **Auto Lock** 
4. retrieve your sample w/holder from auto-sample tray, return holder to countertop
5. close *all* Delta windows
6. log off computer: **Start → Log Off → Log Off**

Hot-Keys, Shortcuts, Tips

1. **Home** key contracts to full, original spectrum
2. **End** key automatically adjusts y-scale zoom so tallest peak fills the window
3. **Backspace** key reverts to previous spectral zoom setting
4. **F1** key immediately brings up **Zoom** side-menu
5. to change spectral line-width:
   A. in the processing list (right-hand-side) click **sexp:**
   B. below the list, in **Width** field, enter desired value
   C. click **GO Process** (upper right-hand-corner)
   - a lower **sexp** value increases resolution but also increases noise
   - a higher **sexp** value smoothes noisy data, but with loss of resolution
6. to change zero-fill:
   A. in the processing list (right-hand-side) click **zerofill:**
   B. below the list, in **Times** field, enter desired value (**4** is ideal)
   C. click **GO Process** (upper right-hand-corner)
   - zero-filling adds imaginary data (zeroes) to end of FID
   - result is a much smoother spectrum, often with a gain in resolution
7. to acquire more scans in same amount of time (for advanced users):
   A. acquire normal spectrum, observe FID, when does it decay to zero?
   B. in next acquisition, reduce **x_points**, observe decrease in **x_acq_time**
   C. in **Pulse** tab, reduce **relaxation_delay** from default of 5 seconds
   D. increase **scans**, acquire
   ** Be careful not to truncate the FID! **
8. for standard ^{1}H-*decoupled* ^{13}C acquisition, choose **single_pulse_dec.ex2**
9. for standard ^{1}H-*coupled* ^{13}C acquisition, choose **single_pulse_dec.ex2**
   - in **Pulse** tab, uncheck **decoupling**
   - in **Pulse** tab, keep **noe** checked for best results
10. for elevated temperature acquisition:
    - in Sample window, click **Turn Temperature ON** icon 
    - input **Target** temperature, let stabilize
    - lock and shim sample as normal
    - when finished, click **Turn Temperature OFF** icon 

    ** Before you exit and walk away, make sure the temperature is indeed OFF **