Quick Summary: 1. Sample into Autosampler (1-3) 2. Select Experiment (#6) 3. Submit (#7) (from submit mode)
4. Process + Plot (from spectrometer mode)

User's Guide to NMR: General

- For help, see Dr. Jasperse, Hagen 407J, phone 477-2230 Draft 10/28/15

Default conditions:
1. Assumes CDCl3 as Solvent.
2. Want ≥0.8 mL of CDCl3 normally
3. Printing on MSUM printer
4. H-NMR

1. Add sample to a Spinner/Turbine

2. Adjust depth by placing the turbine into the golden depth finder

3. Load sample/turbine into autosampler.

- Press the round white **Access Request Button** on the panel below the sample trays/doors
- **Wait until "status" light turns to a solid yellow, and the message panel reads "door unlocked"**

4. **Opening Program on Computer: Usually already open**, and usually to correct "operator"
   - If not open: Operator: your class or research group Password: none.
   - To switch operator, click **Logout** (below main display screen) and select the correct operator

5. **"Submit" vs "Spectrometer" modes:** New Study/Submit Queue to submit; Spectrometer to print/view
   - Spectrometer => New Study: click "**New Study**" button (lower left)
   - Submit queue => Spectrometer: click "**Cancel**" button (lower left)

6. **Experiment Selection** (from within Submit mode). Usually preselected for organic labs.
   - If not already in New Sample/submit queue mode, push **New Study** button on lower left
   - **Proton8** is the normal H-NMR experiment, under the "UserStudies" folder
   - For some classes/operators, **Proton8** has been set to open by default, since most NMR's are regular H-NMR's
   - Add experiments as needed from the Experiment Selector.
   - To **edit or delete**: **right click** on experiment and select "Open Experiment" or "Delete Experiment"

7. **3 Step Submission** (assuming the experiment already specified, and still/already in Submit mode).
   a. Fill **Sample Name** (for both computer filing and printout recognition)
   b. **Click Sample Spot**: Click on the button showing your sample site. (Remember/record! ☺)
   c. **Submit**: clicking the red **Submit** button on the lower left side.
   - **Note**: Can repeat this 3-step sequence for new samples/new students, if running same experiment

   - **Comment box**: (can add comments for the paper printout). (Control C to cut and Control K to paste)
     Other submission options of possible use for advanced labs, research, or offsite Concordia users: **Solvent**; **Autoplot** (offsite Concordia users should turn this off); **Email**; **Email Address** (offsite Concordia users should set this correctly!☺); **Lock**: (with non-deuterated solvent run unlocked), **Shim** (with non-deuterated solvent run 1H PFG); **Tune**

8. **Opening Completed Samples for Printing and Processing. ("Spectrometer Mode" required)**
   - Must be in "Spectrometer" mode, not "Submit" mode. (If already in spectrometer mode, "Study Queue" on left will either display "Location queue" or else "Spectrometer")
     - If in "Submit" mode, "Study Queue" on left will display "Submit queue". Click "**Cancel**" to toggle into "Spectrometer" mode
   - In "Spectrometer" mode, must have "Zones" map displayed (96 sample nodes show). If not, click on little circle icon () to the upper left of the spectra-display panel.
   a. Right click on sample number
   b. Click "Show Study"
   c. Click on file folder name located on the left
   d. Then **double click** on spectrum you want to view to load it into the spectra-display viewscreen.
   e. Process > **Auto Plot** or **Print**. See next page for more detailed printing and processing instructions.
   - Re-click the little circle icon () to get back to zone map in order to open other files
   - To return to "Submission" mode in order to run more samples, click "New study"

Concordia Offsite:
1. Print Preview
2. Enter Correct Email address

9. **Logout:** Click **"Logout"** button underneath spectrum-display

10. Plotting (when wanting non-automatic plots)

- Must be in the process mode. (Highlight "Process" beneath the spectrum display)

a. Click "**Auto Plot**" or "**Print**" button, way on lower right corner of page.
b. Re-click if you want to print additional copies for the other students
   - Note to offline Concordia users: this "plot" command will print to MSUM NMR-room printer. ☺
   - For advanced labs or research groups, additional plot preferences are available in the process mode by clicking "**Plot** (Beneath spectrum display, 2nd from bottom underneath "Start")

11. Horizontal Expansions

- With spectrum displayed on screen, use a panel of display icons on the far right.

a. Click on the magnifying glass icon (6th icon down, )
b. Move your cursor to the left end of the zone you want to expand, then **hold down left mouse button** and slide it to the other end of the zone you want to expand.
   - To return to the full display, you can either click on the 3rd icon () or the 5th icon ().
   - If the lines aren't tall enough, type "vsadj" (vertical scale adjust) on the command line.

12. Manual Integration: Defining Integrals Yourself (see #13 to also give nice integral numbers)

- With spectrum displayed, must be in the process mode ("Process" beneath the spectrum display)

a. Choose "**Integration**" (Beneath spectrum display towards left, 2nd underneath "Start")
b. Hit "**Clear Integrals**" button (slightly further to the right and lower down from previous button)
c. Hit "**Interactive Resets**" button (immediately above the "clear integrals" button) and define
   1. Move cursor beyond the left end of the signal you want to integrate.
   2. Left-mouse click-and-release
   3. Move the cursor to the right of the signal, and again click-and-release. Everything between the two "clicks" will be integrated.
   4. Repeat this for each area you want to integrate.
d. Click very top cursor icon () to the right of the display screen to regain normal cursor function

13. Setting Nice Integral Numbers (While already in integration mode following steps a-d above**)**

a. Click cursor on one of your integral regions
b. Click "Normalize Area to" "**Single Peak**" below "Set Integral Area" panel underneath the display
c. Set "integral area" to some nice whole number (1, 2, or 3, depending on your molecule)
d. Click the "set integral value" button
   - If it says "cursor is outside of integral region", then reset the cursor on an integral of choice, and re-click the "set integral value" button again.
   - Click "**Auto Plot**" (lower right) in order to print.

14. Other Processing Options for Advanced Users/Research Groups/2D-NMR

1. Peak Picking
2. Vsadj
3. wp=2p sp=2p plot
4. Insets
5. Arraying spectra
6. Absolute Concentration Integration
7. 2D NMR processing, including varying the signal intensity

15. Opening Spectra From the Data Folders

- Click on the Folder icon and find your class or research professor's folder
- Double-click on the folder with your name.
- Double click on the experiment file
- To get the Folder icon to go back up a step, click on the Folder icon again, then click ONCE only on the little icon that shows an arrow up

16. Getting the last sample out and replacing with a Lock Sample (if auto-eject isn't turned on)

a. In "Spectrometer" mode, display "zones" map ()
b. Right click on sample 48 => select "Sample in Magnet" (3rd choice from the bottom) => OK.

17. **Logout:** Click **"Logout"** button underneath spectrum-display