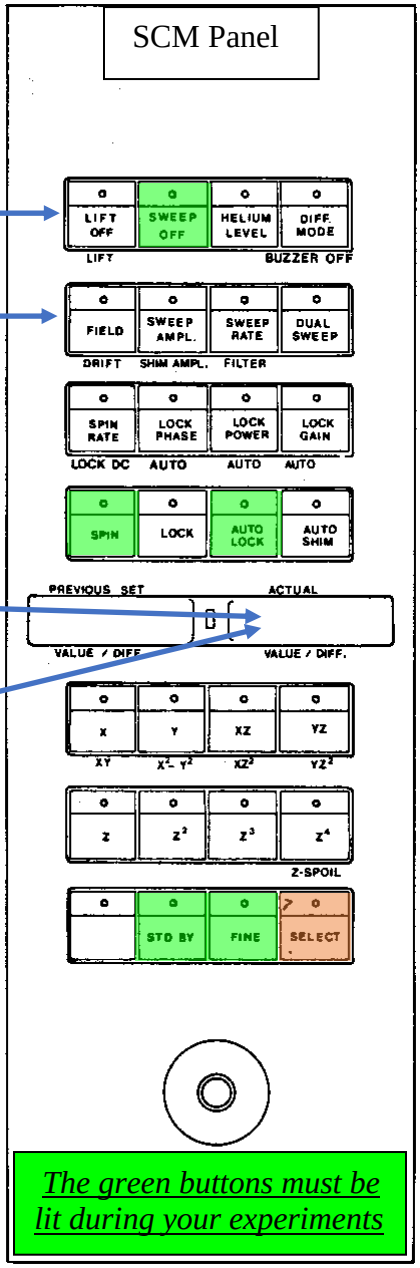
CHEM 445 - NMR Lab Instructions

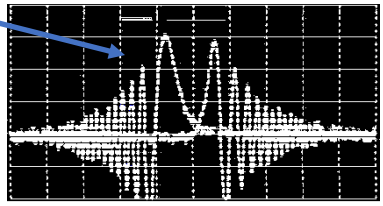
1. Log into Windows and start the NTNMR program.

2. Thoroughly clean your sample tube using a Kimwipe.

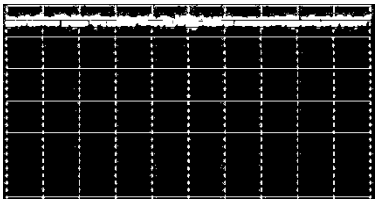
3. Eject the standard sample using the SCM panel:
   a. Remove the cap from the magnet.
   b. Press the orange **SELECT** button and then **LIFT**.
      (You should hear air flowing and the standard sample will be ejected from the magnet.)
   c. Take the water sample out of the magnet and remove it from the spinner turbine. Insert your own sample into the spinner and adjust the sample height using the plastic gauge.
   d. Insert your sample with spinner into the magnet.
   e. Press the **LIFT OFF** button.
      (Your sample will slowly drop into the magnet.)
   f. The sample should be spinning with 15-20 Hz.
      If the sample won't spin, clean the tube again.



4. Press the **FIELD** button and use the wheel to display "**1700**".
   If you don't see the Lock Signal on the black window yet, you will have to search for it by increasing or decreasing the **FIELD** value until the Lock Signal is centered in the window.



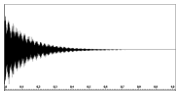
5. Press the **AUTO LOCK** button.
   Wait until all lights have stopped flashing and the lock signal has stabilized at the top edge of the black window.



6. Now the magnetic field needs to be "shimmed":
   Press the Z button and turn the wheel.
   The goal is to make the lock signal go as <u>high</u> as possible.
   Repeat with Z^2. Optimize Z and Z^2 several times.

7. <u>In NTNMR</u>, open the default Proton NMR parameter file for your solvent, i.e.: *1H-water.tnt*
   To observe other nuclei, open instead: *13C-water.tnt, or 59Co.tnt, or 55Mn.tnt, or 67Zn.tnt*

8. Click ZG to record the FID:
   (Free Induction Decay)



^{1}H will finish after ~2 minutes, ^{13}C after 1 hour.

9. Use the ***File | Save as*** dialogue to save the raw data as: "ChemXXX-Name-Sample" (use your own course number, last names, and sample name)

10. ^{1}H only: Process the data using: Ctrl-B (baseline fix), Ctrl-F (Fourier transform), Ctrl-H (Phasing). Other nuclei: Process the data using: Ctrl-B, Ctrl-E (exponential apodization), Ctrl-F, Ctrl-H.

11. Again, use the ***File | Save as*** dialogue to save the processed data, this time as: "ChemXXX-Name-Sample-processed"

12. *Calibration* of the chemical shift reference
    - Expand the solvent signal, using a click-and-drag operation of the mouse.
    - Left-Click on the reference peak (TMS or Solvent).
    - Right-Click anywhere in the spectrum and select ***Processing | Set Reference***.
    - Select **ppm** units and enter the correct chemical shift. (See the solvent table.)
    - Press Ctrl-S to save.

13. *Peak picking*
    - Select ***Option | Peak Pick***.
    - You can adjust the height of the black horizontal bar that will appear. All peaks that come out of the black area will be picked.
    - Click on **Apply** when done. The program will only number the peaks at this point. Later, before you print, you can decide what the labels should be (i.e. ppm).
    - Zoom into the spectrum and delete or add other peaks. Click **Exit** when you are done.
    - Press Ctrl-S to save.

14. *Integration (only needed for ^{1}H spectra)*
    - Select ***Option | Integrals***.
    - First, display the complete spectrum and click on **Remove** to erase any pre-existing integrals.
    - Click-and-drag the mouse over the signal you wish to integrate. (Expand, if necessary)
    - Right-click in the highlighted area and select **Add Integral**. Repeat for other peaks.
    - Double-click on any one of the integrals. Set the **Active Integral** to select the integral you wish to normalize.
    - Set the **Assigned value** to the desired value. Click **Apply** and then **Close**.
    - In the left panel change the labels to **Assigned Value. Exit**.
    - Press Ctrl-S to save.

15. *Scaling your spectra*
    - Select ***Option | Amplitude Adjustment*** to define the size and width of the printing region.

16. *Printing your spectra*
    - Use the ***File | Print Layout*** dialogue to select and modify the settings for the printout.
    - Uncheck **Parameters, Line Fit** and **Logo.**
    - Check the box **Integrals** and under Settings check **Show Value.**
    - Check the box **Peak Picks** and under Settings select **Label by chemical shift.** Set the **Precision** to **2** or **3** (digits after the decimal point)**.**
    - Enter or change the title for the printout under Comment Settings, if desired.
    - Grab the scale and drag it all the way to the bottom.
    - Click on the printer icon to print out the spectrum.