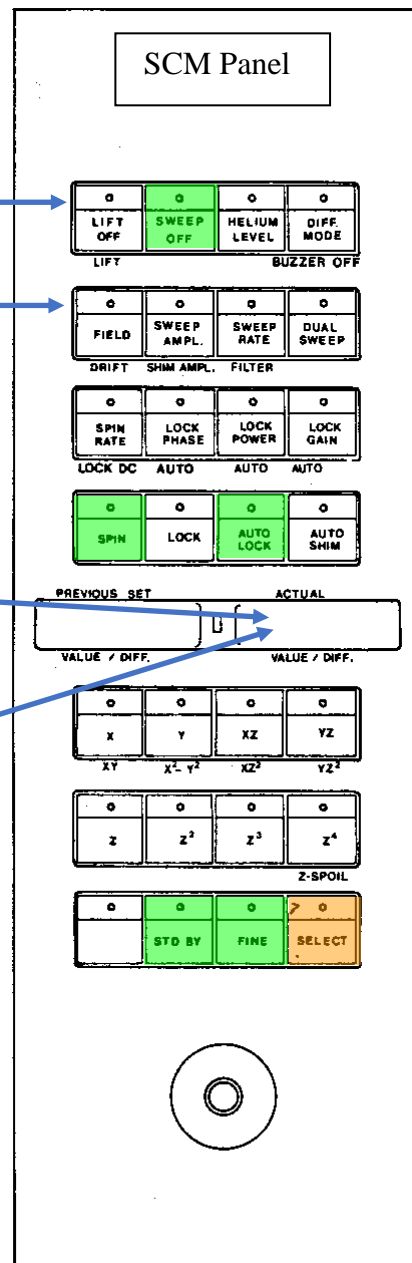


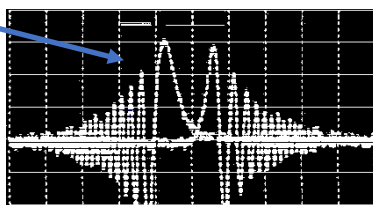
CHEM 445 - NMR Lab Instructions

Sample Handling – Magnet Adjustment

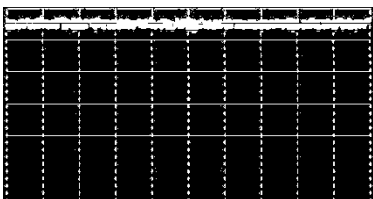
1. Log into Windows and start the NTNMR program.
2. Thoroughly clean your sample tube using a Kimwipe.
3. Eject the standard sample using the SCM panel:
 - a. Remove the cap from the magnet.
 - b. Press the orange **SELECT** button and then **LIFT**. (You should hear air flowing and the standard sample 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.
 - 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 "1100".
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 line rise as high as possible. Repeat with Z². Optimize Z and Z² several times.~~
(Skip this step, automatic shimming is now included in the automation scripts!)

The green buttons must be lit during your experiments

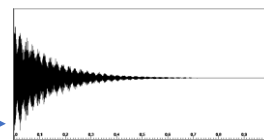
NMR Data Acquisition with NTNMR

Now you can record and process your NMR spectra using NTNMR's scripts:

1. Open the default Proton NMR parameter file for your solvent, i.e.: 1H-DMSO.TNT
(Make changes in the dashboard as needed, i.e. receiver gain, pulse width, ...)

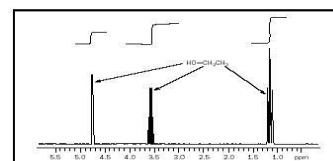
2. Click: <SCRIPTS><HNMR>

Message windows will indicate the start and the end of the experiment.
It will take approx. 5 minutes to optimize the magnetic field homogeneity ("shimming") and to record the proton NMR data:



3. Use the **File | Save as** dialogue to save the raw data as: "ChemXXX -Name-Sample-1H"
(use your own course number, last names, and sample name)

4. Process the data using: <SCRIPTS><PROCESS>



5. Use the **File | Save as** dialogue to save the processed data as a separate file: "ChemXXX-Name-Sample-1H-processed"

Spectrum Processing with ACDNMR




6. Start the ACDNMR program and open the processed file.

7. Click **Phase** and try the **Auto** buttons or use **Mouse Ph** for manual phasing. Press  to finish.


8. Calibration of the chemical shift reference:

- Expand the solvent signal, using a click-and-drag operation of the mouse.
- Click **Reference** and Left-Click on the solvent reference peak. Select your solvent from the list.

9. Baseline Correction, Peak Picking, and Integration can be done automatically:

- Click **Baseline** - **Auto** - **Result** - 
- Click **PeakPick** - **Auto** - 
- Click **Integrate** - **Auto** -  (Use manual integration for better accuracy.)
Adjust the "Reference" number to obtain reasonable integral values.

10. Create a PDF file for your report:

- Click  to create a PDF file.

- Adjust the ppm values in the "Spectrum Region" field.
(using standard regions will allow visual comparison of spectra,
i.e. 11.0 to -0.5 for proton spectra and 220 to -20 ppm for C-13)