NMR New User Workshop

First session

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Notes reproduced from Dr. Weiguo Hu

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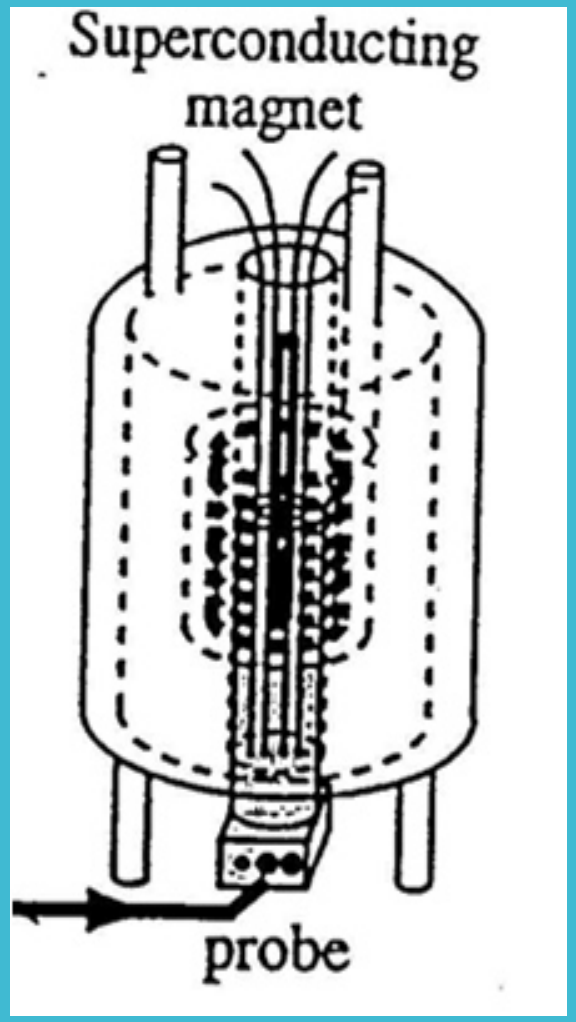
Booklet:

https://udrive.oit.umass.edu/weiguoh/nmrhandouts/Introduction-NMR.pdf

Magnet Safety



- Very strong magnetic field!
- ca. 100,000 times stronger than earth magnetic field

- Pregnant women should stay away
- People with pacemakers should stay away

Magnet Quench

Quenching is the process whereby there is a sudden loss of superconductivity in the magnet coils, so that all the electric energy in the coils becomes heat and suddenly boils the liquid helium, causing a loud “boom” and people in the room will experience difficulty breathing. You should leave the room immediately when this happens!

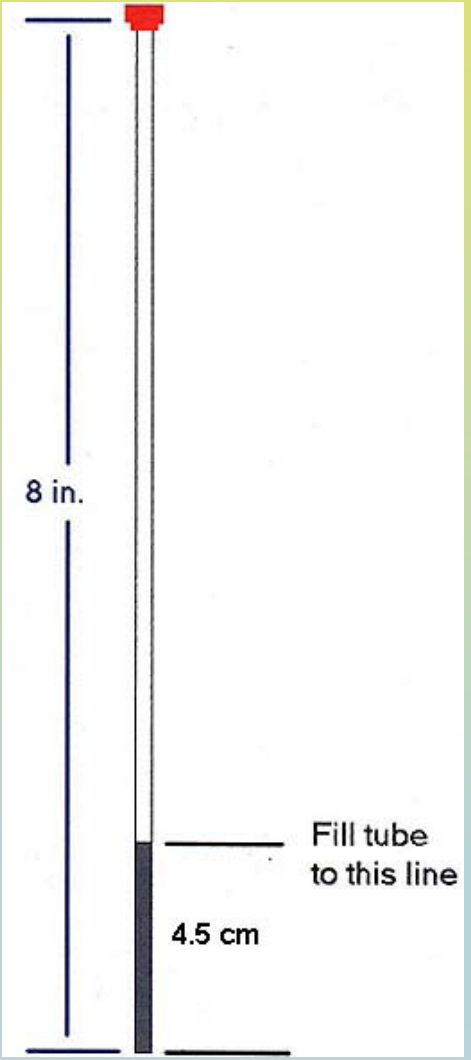
– Never get iron object close to magnet

- Wrenches, heat gun, etc could fly to the magnet
- The “bang” would **quench** the magnet

Keep credit cards, cell phone, watches, etc. away !

Sample Handling Safety

- **NMR tubes are brittle; handle with care**
  - Breaking sample tubes are frequent events!
  - Select a spinner that fits the tube properly
  - Stuffing kimwipes to fit the tube in spinner is NOT Okay !
- **Common mistake**
  - Insert tube without spinner
- **Measure the correct sample depth**
  - Incorrect depth makes lock and shim difficult
  - too much depth will get the sample stuck
  - If you have less than 4cm sample, center your sample around the thin black line rather than push it to the bottom



What to do if you have insufficient solvent?
https://blogs.umass.edu/weiguoh/

Choices that you have

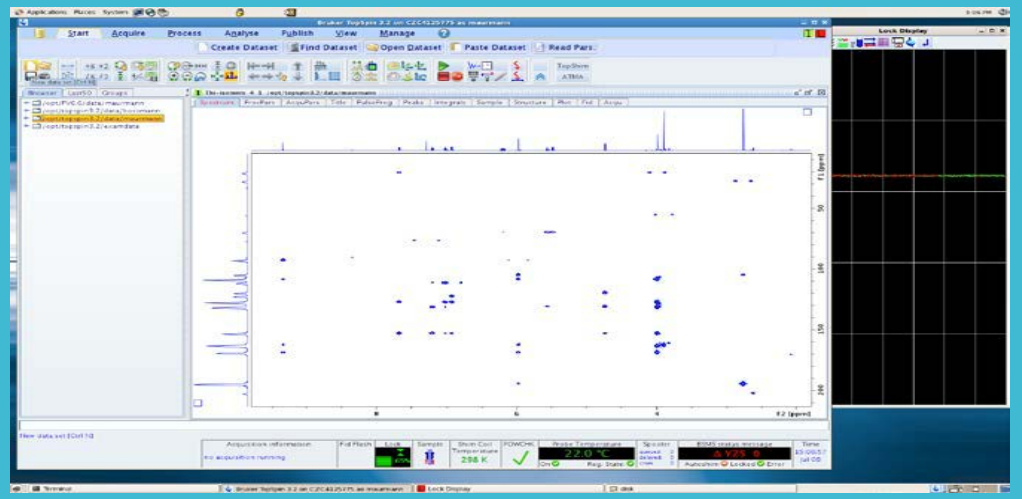
- **Icon NMR**

–Multiple samples

–Multiple spectrum

–Undergraduates

- **Manual mode**



–More choices and flexible
–Faster

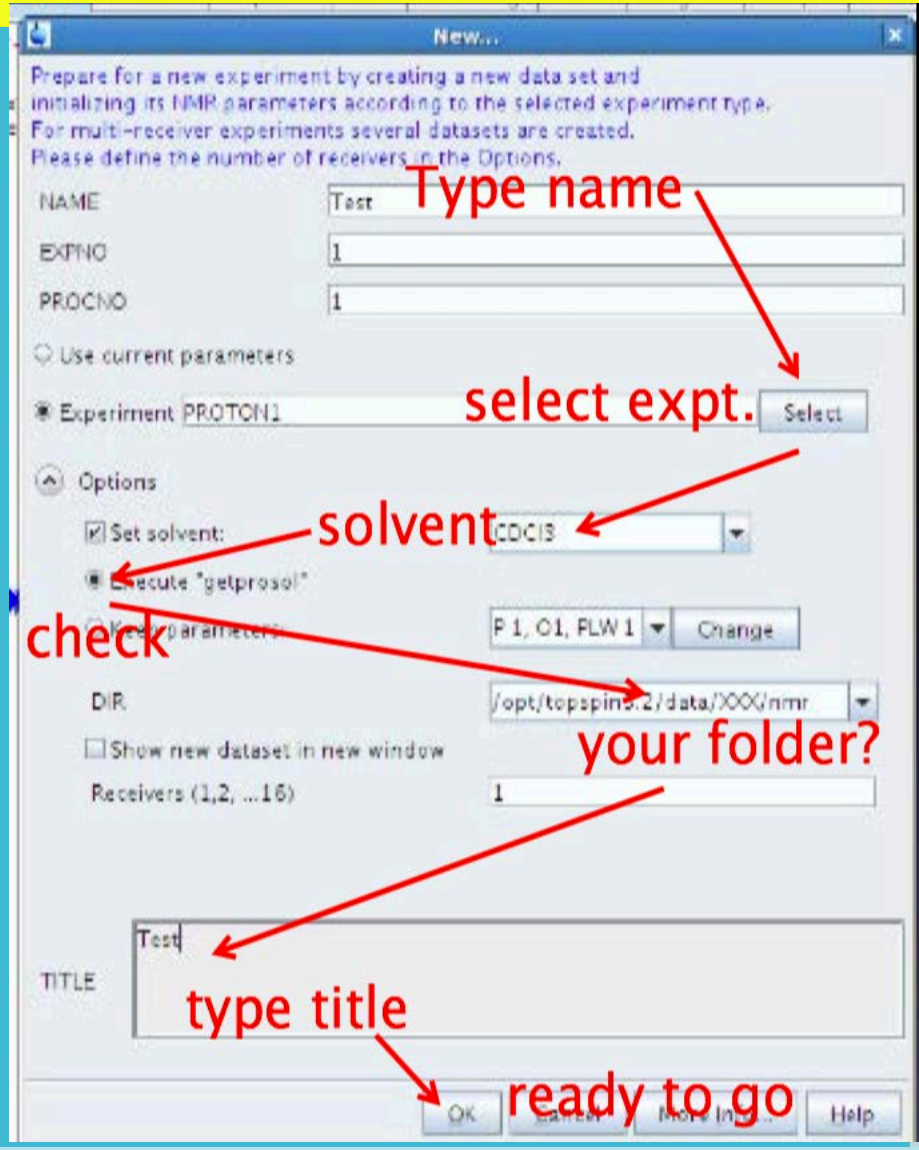
Preparations

1.Inject your samples :
- sx #

2.Create new file: you must find the correct template file
- edc
- check get prosol, and see if the DIR is directed to your folder

3.Tuning the probe frequency to what your are going to detect
- atma

4.Load a generally good shim set.
- rsh shims.best



Shimming and Locking

Shimming: to eliminate inhomogeneities in magnetic field

– the magnetic field is far from homogeneous initially

– the sample become slightly magnetized and create additional inhomogeneous fields.

– the process of correcting for these inhomogeneities is called shimming the magnet

Locking: to compensate for the slight drift of magnetic field

- If the magnetic field strength changes during the course of the acquisition, the signals will appear broadened and the spectrum will have poor resolution.

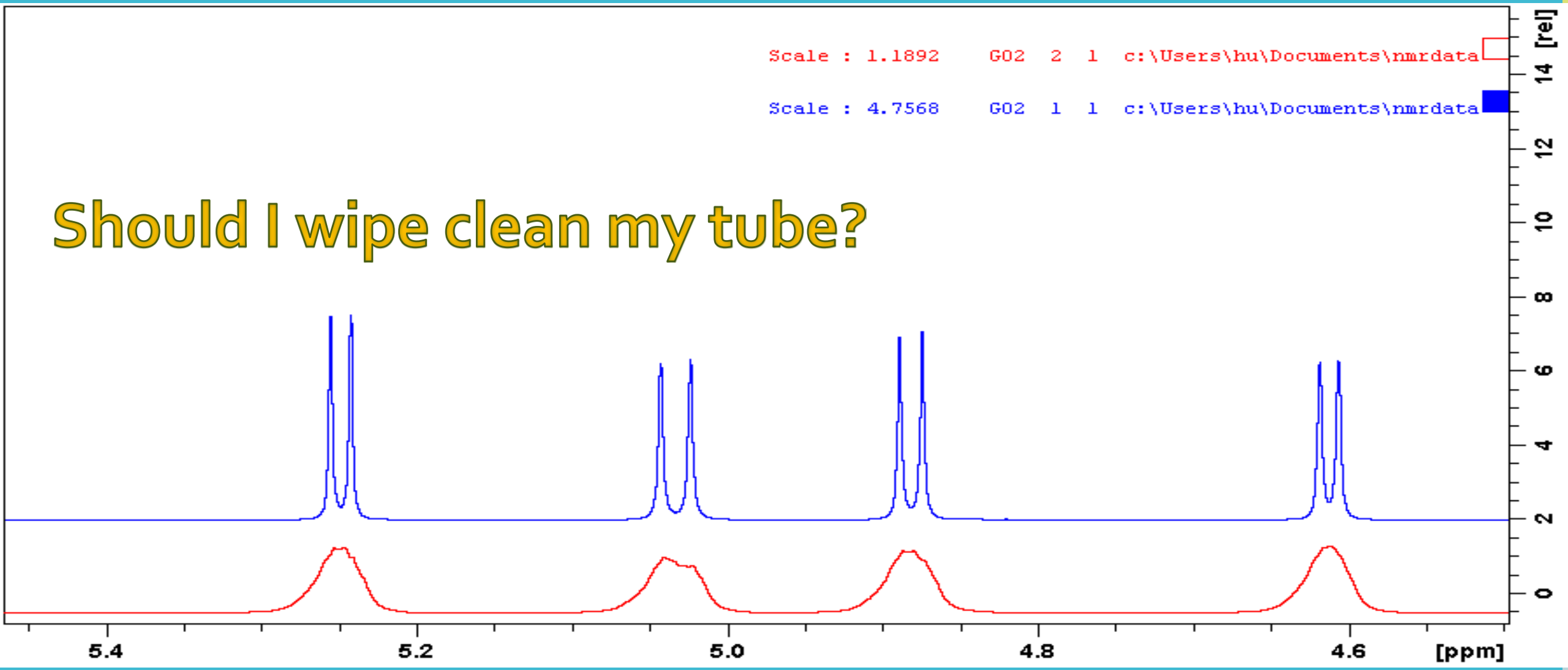
– use 2H signal

– This is one reason why we need deuterated solvent to do NMR!

– "**lock [solvent name]**" also calibrates chemical shift

Stable and homogeneous

Shimming and Locking



Stable and homogeneous

Important Factors That Affect Shimming

rsh shims.best reads in the most recent good shimming parameters
– However, best shimming condition is sample dependent, so you still need to touch-up shim for each sample yourself.

Spinning is optional. Gives better resolution for simple 1D.
– Turn off spin when running 2D

Magnetic susceptibility – everything becomes magnetic in a big magnet!
– Sample homogeneity: insoluble particles, dirt stains on tube wall
– Quality of tube
– Shim would change significantly If you switch between non-polar and polar solvents

Gradient shimming (topshim): directly detect coefficients

How to manual shim?
https://blogs.umass.edu/weiguoh/

Running Experiments

rga sets receiver gain (rg – just like the volume knob in your radio).

zg will start the experiment

ns number of scans.

tr transfer data to hard disc without stopping expt

halt stop expt and save data

stop will stop expt immediately and discard data.

efp Fourier transformation (with optional line broadening and phase correction)

Processing functions: feel free to play with all the buttons

http://www.youtube.com/channel/UCZlxixehE9JONeGTVqMjEjw

Phasing; Baseline Correction; Integration

Phasing is the "p" in efp

– apk and manual phasing (0^{th} and 1^{st} order)

Baseline must be corrected before integration

– abs is usually adequate for routine uses

– To integrate small peaks on the shoulders of big peaks, manual baseline correction (bas) is necessary

Integrations of 1H spectra are crudely reliable

Peak heights are not a good measure of population

Phase Correction

Phase distortions cannot be corrected by baseline correction

apk often does not do an ideal job

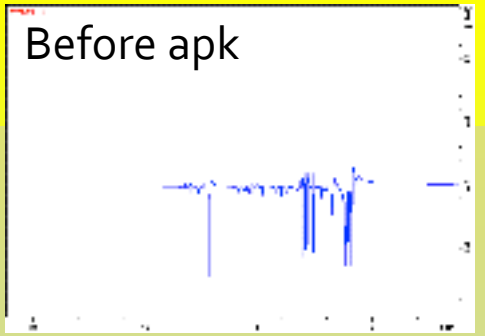
Manual phase correction:

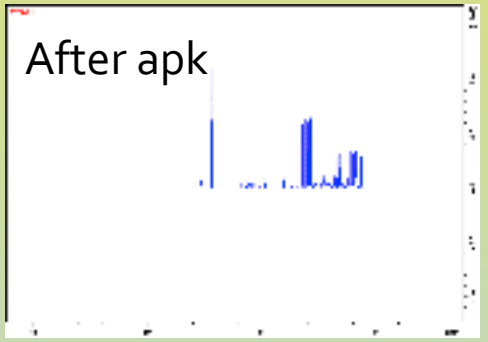
0^{th} order: correct phase of all peaks to the same degree

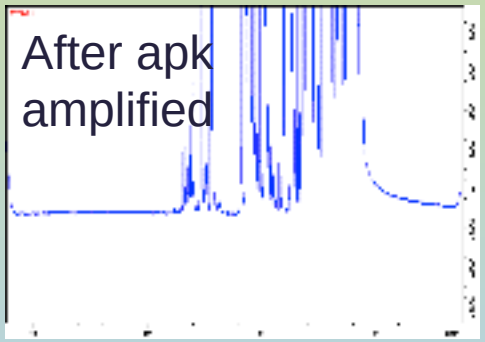
1^{st} order : correction amount ∝ distance to the pivot line

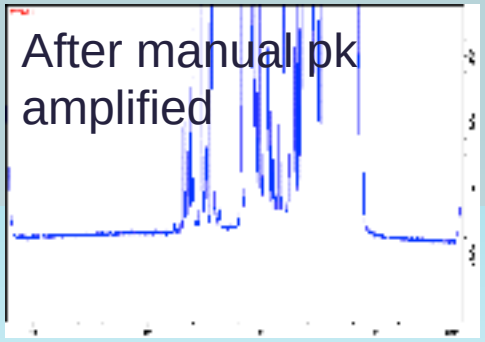
Objective of phase correction:

to make baseline visually continuous so that baseline correction can work (baseline correction requires a continuous baseline)









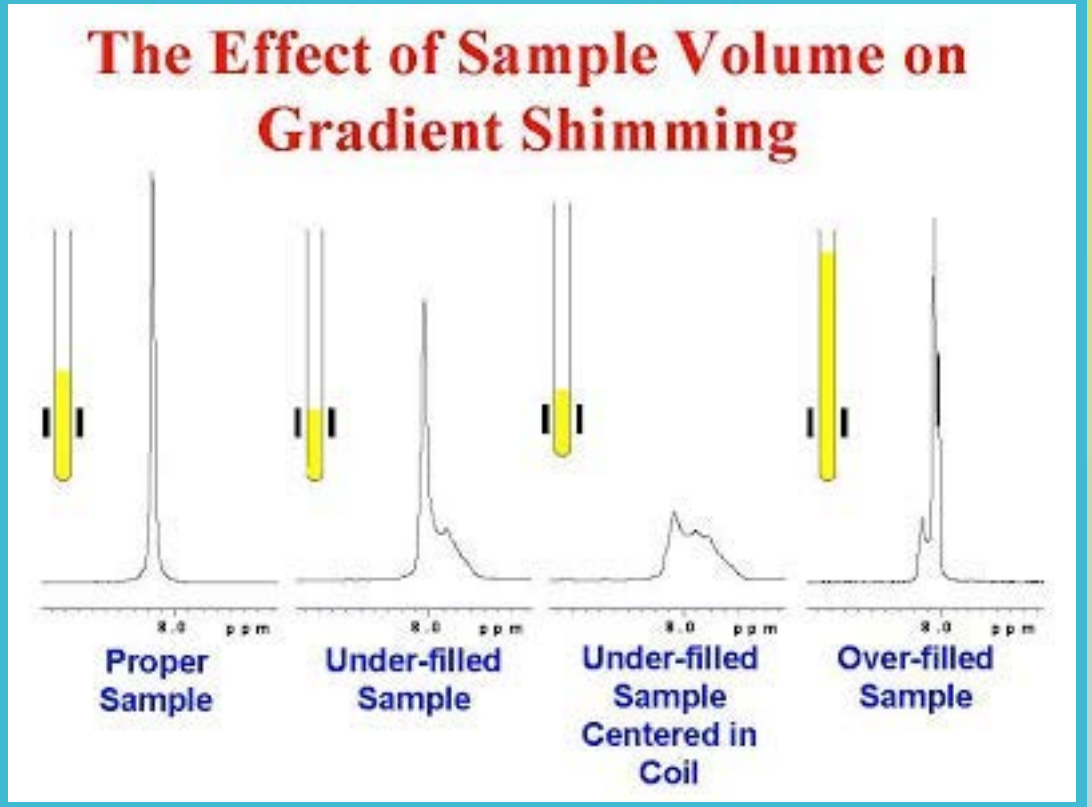
Outline of Procedures

Remove wallets, watches, keys. Keep steel objects far from the magnet

- Check the blue LED light on the cryo-plateform (Must be solid on)
- Insert your sample: sx #
- Create new file: edc
- atma; rsh shims.best; lockdisp
- lock; select solvent
- Shim (manual (bsmsdisp; several iterations of z and z2) or topshim
- rga; ns; zg; efp
- Phase correction: apk
- Baseline correction: abs
- Integration; transfer data out
- Get your sample back : ej
- Logout (not logged out promptly will result in excessive charges)

Common Experimental Problems

- Difficulty locking:
  - Bad shimming (2H signal is broad and low)
    - Did not do rsh shims.best
    - Sample depth not correct
    - If less than full sample, did not center sample around black line
  - Instrument may be “Fatigued”… try typing the command again
- Spectrum has a poor resolution:
  - Indications of a shimming problem:
    - Every peak has the same peak shape
    - Peak shapes are often asymmetric



A brief introduction to NMR
https://blogs.umass.edu/weiguoh/

How would you explain NMR to a non-scientist?
https://blogs.umass.edu/weiguoh/