Obtaining ^{1}H NMR Spectrum

After lock and shimming, type:

nt=8 d1=2 ga (crude NMR)

nt=8 d1=8 ga

- Larger dl gives better integral by increasing relaxation time
- Larger nt gives lower noise by increasing the number of scans

Working up spectrum

Type:

1. wft dc vp=12 aph f cz vsadj

wft = show spectrum
dc= data line correction
vp=12 = vertical position
aph= autophase
f= show
cz= resets integrals
vsadj=adjusts peaks to fit in spectrum

2. reference solvent peaks (i.e. $CDCl_3$)

- Right click on solvent peak
- Type nl (nearest line)
- Reference peak in analyze tab/default
- Type 7.26 ppm (not Hz) and press 'enter'

3. save file

- Type 'svf'
- Type the file name following proper format (i.e. initals-book-page-a)
- Press 'enter'

4. phase peaks

- Increase peak intensities (display - +)
- Ensure that the baseline is flat and no dips are observed
- Click phase button, if needed, until baseline is flat

5. baseline smoothing

- Click on 'integral' icon
- Cut (scissors icon) integrals for *every* peak above 3:1 above the noise (solvents, compounds etc.)
- Type: 'bc' enter
- 'cz' enter
- 'vsadj' enter
- baseline should be flat (do NOT type 'wft' or else smoothing function will be lost)

6. integrals and integration

- cut integrals for desired peaks only (excluding solvent peaks)
- after all integrals are set, integrate the peak with the lowest value (i.e. if one peak integrates to one proton, set the integration value for that peak to '1.0')
- 'dpir' displays integrals on the spectrum
- click icon to either select one peak or to view a range in your spectrum to exit mode

7. printing

- set your spectrum to a ppm range to 9.05 to -0.5 ppm
- this range should be the same range for all of your nmr spectra (except when the peaks are outside of this range)
- PRINT: type ' pl pap pscale pir page'
  pl=plot the spectrum
  pap= print parameters
  pscale=print scale
  pir=print integrals
  page=print
- Magnify regions to look at more closely, then type:
- 'pl pltext pscale pir ppf page'
  pltext=print label
  ppf=ppm label for each peak integral
- increase peak intensities if needed
- do not print more than 50 peaks per page