Nuclear Magnetic Resonance (NMR) Spectroscopy Protocol

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Henry Han

Interpretation

For standard 1H NMR spectrum, there are three pieces of data we would like to see:

(1) **Peak position**: Chemical shift (in ppm) of each peak
(2) **Peak Area**: integration
(3) **Peak "Splitting"**: coupling (e.g. doublet, J = 2.0 Hz)

Pre-NMR Steps (Organic Synthesis)

1. Obtain a clean, small 50 mL Round Bottom Flask (RBF), and weigh it **before** putting anything inside to determine the product yield. Be careful of weighing because of small yields.
2. After TLC, combine all fractions with same compounds into the RBF. When combining fractions, rinse the original container with small amounts of solvent and add the rinsing to the RBF.
3. Rotovap the combined fractions.
4. Weigh the RBF again to determine the yield for each product.
5. Dissolve sample in ~0.5 mL deuterated solvent ($CDCl_3$). Sample shouldn't be too much (diluted) or be too little; around three fingers in height in the tube is sufficient.
6. Carefully transfer sample to a clean, dry NMR tube. 3 fingers of liquid. Cap, **label**, and take down to the NMR room. Do not invert tube!
7. Before going to the NMR room, check SciFinder database for similar spectrum – come prepared!

NMR Setup

1. Log in with jrscheerer, or jrsche, password: Chemi$try42
2. **e** – eject sample already in the NMR, then use kimwipe to touch turbines and transit samples you prepared.
3. **i** – insert sample.
4. Enter sample name (initials, book number, page number, crude/pure, fractions in sample). Example: HH_kenone_frac22_23_10_13_2017. Put name under **start** tab. In the **sample name** space, and **comments box**.
5. Select solvent $CDCl_3$ or proper solvent being used.
6. Lock: go to lock, unclick the checked box next to lock, click **find z0**.
7. Shim – **gradient shim**, the good number should be at least around 30-40.
8. Select experiment: proton NMR or carbon NMR. To load the experiment, drag the correct experiment from the left-hand options bar into the window. Set nt (number of transients) and bs (block size) - **nt=32** and **bs = 4**. (nt=3200 for carbon)
9. **ga** – start the experiment.

Data Processing (H MNR)

1. Type commands: **wft aph vsadj cz f**
   wft – fourier transform and view spectra
   aph – autophase spectrum
   vsadj – vertical scale adjustment
   cz – clear zeros for integrals
   f – full spectrum (-2 ppm to 15 ppm)

2. Spectrum reference: standard reference peak in $CDCl_3$ will be at 7.26. Click ~7.26 and type **nl** to select the nearest line, and then type **rl(7.26p)** to set the reference line. Reference TMS peak at 0 ppm also.
3. Integration:  Full Spectrum icon – turn on **integral** and use **cutting tool** to chop up into appropriate pieces. Choose a peak with known integration area, go to the integration tab, and enter the number (known area) where it says integral area.
4. Set the size of the spectra by typing **sp=-0.5p wp=9.5p vsadj →jrsstd**

Print and Save

1. Go to auto plot preview and print. Print parameters in horizontal box: click drop down menu by display parameters and select horizontal box. Or pl pscale ptext page
   pl – print line
   pscale – print scale
   ptext – print associated text
   page – actually prints your spectra
2. **svf('filename')**
3. **e** (eject sample) and **i** (insert the standard $CDCl_3$).

Other

1. if NMR is not responding, and won't abort when type **aa**, go to **terminal** under **applications**, type **su acqproc** and retype it when something appears and type **exit**.

Post NMR

1. Use DCM to rinse glass pipet used to transfer sample back into vial or sample container
2. Pour contents of NMR tube back into sample container
3. Rinse NMR with DCM and deposit rinses in sample container
4. Rotovap sample back down
5. Rinse NMR tube at least 3x with acetone and put back in oven

Getting NMR Spectra Off the Computer

1. Open NMR software
2. Find and open your **saved.fid** file on the little window on the left side of the screen
3. At the top of the screen, click **Utilities** → **Printers** → **Plotter** drop down menu → Select "**eps**" → OK
4. Type: **pl pscale page ('YourFileNameHere.ps')**
5. If you want to get the chemical shifts too, you can type **pl pscale ppf page**…
6. Make sure you type the parentheses and apostrophes
7. Go to desktop and open the folder for dyoung
8. Find your **.ps** file, and copy/paste to your flash drive
9. At your computer, convert files from postscript files to pdf: http://ps2pdf.com/convert.htm