Beaudry Group Routine ^{1}H NMR Guidelines

1. As scientists, it is important that we commit to the collection of first-rate data for all of our reactions. Furthermore, it is equally important to put into place an archival system for this data to be published, scrutinized, resurrected, analyzed by us and others years down the road. Thus, we archive data using a system others can understand, not individual case-by-case systems.

2. Please name all of your NMR files by your initials and the corresponding notebook page. For example, CMB2145 would be Chris Beaudry's NMR from notebook 2, page 145.

3. Be sure that you change the solvent parameter before acquisition to indicate which solvent you are using. This is especially important if you are using C_6D_6, since you can lock, shim, and acquire with the $CDCl_3$ settings and get a residual solvent singlet in a similar chemical shift range.

4. Print your routine ^{1}H NMRs from 10 ppm to -1 ppm. This is so we can overlay print outs. In the event that resonances appear downfield of 10 ppm (say an aldehyde), stick to printing an 11 ppm window. For example if your aldehyde has a singlet at 10.5 ppm, then print from 11 ppm to 0 ppm. In the rare event that you have resonances spread across more than an 11 ppm range, then print in whole number ppm ranges that make sense. For example, 12 ppm to -2 ppm.

5. Keep electronic backups of all your NMR files. Keep paper copies organized in a binder.

6. Please use the following extensions for your files:

SM – NMR of the starting material

X – NMR of the crude material

1,2,3 etc. – NMR of different purification fractions in <u>order of isolation</u>.

A,B,C – NMR of time course experiments

Beaudry runs an experiment in an NMR tube by dissolving the sample in CD_2Cl_2 and checking the purity and shifts of the starting material (CMB2145-SM). He adds a reagent and checks the reaction after 5 minutes (CMB2145-A), 1 hour (CMB2145-B), and 24 hours (CMB2145-C) at which time the starting material is consumed. He works up the sample with $NaHCO_3$ solution and extracts with EtOAc and concentrates the mixture. He then checks the crude material (CMB2145-X) and chromatographs the product isolating a higher R_f impurity (CMB2145-1) and the desired compound (CMB2145-2).

6. In order to publish, we need NMR spectra that are well shimmed, have a good signal-to-noise ratio, are properly integrated, peak-picked, and have no resonances from impurities or residual solvents like EtOAc and Et_2O. To get publication quality spectra, it is okay to rotovap a sample down from $CDCl_3$ to remove EtOAc peaks (do NOT do this before calculating yield). See also the JOC guidelines for publication in the ACS journals.