Notes on Characterizations (NMR, IR, MS) for CH 463/H Project 1
April 20, 2009

More on NMR characterization:

You are expected to use a suite of 1 and 2 DNMR experiments to prove the structure of your benzophenone. To prove the structure you are required to interpret each NMR experiment and write about the analysis in report 1 as part of the characterization of your compound. For Project 1 Report 1, in addition to the NMR, you are expected to run and report good quality data for: R_f from tlc; IR; MS. Compare to published literature as much as possible.

Before running NMR, IR and MS, purify your compound to a white crystalline solid. Prove it is clean by measuring a sharp mp (no more than 2 degree melting point range) that agrees with literature and determine the Rf from tlc and record mobile phase composition.

For initial HNMR and CNMR (if time): Check out an NMR tube from the issue room. Prepare a pure sample (see below). After you have a sample prepared, setup a time with Jo, Nathan, or Somnath who will help you to run the HNMR on the 400. For the 2D experiments (HSQC, COSY and HMBC) submit the same sample and Rodger Kohnert will run these and you will get the pdf's in a day or two.

Prepare the NMR sample by massing 20 +/-5 mg in a small vial. Use a balance to weigh out the sample, don't just "eyeball it". Add about ¾ mL of $CDCl_3$ to the vial and dissolve. If this doesn't work, use a different deuterated solvent, ask a TA. Immediately interpret the HNMR and decide whether or not the sample is good enough to run the other NMR experiments, if not discuss strategies for further cleanup with instructors. For example, if your sample is not clean in the NMR, recrystallize it, dry it and check the new mp, and run the HNMR again. If your sample is now pure, submit the same NMR tube that you ran for HNMR to Rodger Kohnert who will run the suite of 2DNMR experiments and you will get the results back the next lab day. Tag the sample with your last name and first initial and indicate: compound name, structure and request the CNMR, and 2D suite of CoSy, HSQC, and HMBC. All spectra will be saved to pdf format so that you can zoom, etc to analyze and this makes available a convenient electronic format to add to the report 1. These files will be put on the folder at T:chemistry/share/CH 463 2009 using your last name and first initial for file names. As long as you are logged into the science server, you can retrieve the files. Compare the H and CNMR to those in the literature if you located these.

More on IR.

You are expected to include an analysis of the IR in your report 1. The two standard methods of sample preparation for the IR are: (1) make a KBr pellet

and/or (2) deposit a few drops of a solution of the ketone in a volatile solvent on a KBr disc, then evaporate the solvent to produce a thin film of your compound on a KBr disc. Report the major IR bands and use these to help prove the identity of your compound. For example, report the carbonyl stretch; the strong ring+carbonyl str indicative of the diphenylketones; the aromatic CH stretch and saturated CH stretches (not everyone will have the sp^3 CH str); if an ether substituent, report the C-O str; if a halogen substituent report the C-X str. If you have an IR from the literature, compare the major transitions you observed to those reported in the reference.

More on MS.

You are expected to include an analysis of the MS in your report 1. To make a solution to inject in the GCMS, dissolve about 2 mg in 1 mL of DCM and inject 1 uL. Report the M^+ ion(s) and the base peak. Include any fragments that help to prove the identity of your compound. For example, some important mass peaks to discuss might be:
halogen isotope signature; M-X apparent?
loss of CO: M-28;
arylO^+ (one for each ring);
if one nitrogen containing substituent, odd M^+; M-46; M-30; M-58;
loss of M-CH3, M-C2H5 for ethers; continued loss of CO
isotope signature for Cl, or Br