Report 3 Guidelines for Written Portion Project 2
CH 362 & 362H
Winter 2009

The written portion of Report 3 is due Wednesday, March 18, by noon. Please submit your report directly to one of the course instructors or bring it to the fourth floor lab at noon.

Remember there are three parts to this project: (1) the keto-enol solvent study; (2) the metal acetylacetonate complex study; (3) the 1 and 2 D NMR and MS experiments for the unknown. Submit one journal page (about 1 or 2 pages of text or 300 words) of thoughtful proof-read discussion on each of the two studies that you did not present. For the study that you (will) present, please submit an abstract on line (see course web page for link). There are Supplemental Materials Summary report forms that are designed to help you cover the basic points for each project (see link on the course supplemental materials web page).

A thoughtful discussion includes a succinct but informative overview of the problem that was studied, and an analysis of the results explained in scientific terms and within the framework of accepted theories. Of course, you may find it necessary to adapt current theory to describe your results and this is okay as long as any extensions you make are well founded and substantiated.

The first study is the keto-enol tautomerization of 2,4-pentanedione as studied in a variety of deuterated solvents on the 400 MHz NMR. Investigate how the values for Keq from the class data on the web compare with one or more solvent parameters (also posted on the web page and an appendix in the lab manual). What solvent parameter(s) correlate(s) best with the set of measured Keq values found in different solvents? See the lab manual for a discussion of this project and it was covered in lecture. Recognizing that there are differences between solvent-keto interactions and solvent-enol interactions, try to rationalize why the enol form relative to the keto form appears to be stabilized better by certain of these solvents and not others. Discuss reasons why you think the observed trends can be explained by invoking the definition of the correlated parameter.

A second study involves the preparation and characterization of a metal acetylacetonate complex (M-acac). As is typical with any synthesis, there is the synthetic route, the conditions, the yield, and general characterizations of product, such as melting point and IR. In addition, two other experiments are done to uncover information about the bonding in the newly formed complex. *First*, what changes in characteristic vibrations for functional groups are apparent between the spectra for the ligand (acetylacetone) and the complex? If there are differences in the IR, how might one use this information to characterize the bonding between the metal and the acetylacetonate ligand? Next compare and contrast the IR for the metal complex solution (solvent subtracted) with the IR from the KBr pellet. *Second*, NMR allows assessment of the paramagnetic susceptibility, which leads to determining a value for the number of unpaired electrons for the metal in the complex you synthesized. Some of the questions that you might pose answers for in your discussion are: what does the comparison for the experimental value for

n indicate about the degree of covalent character for the metal to ligand bonding found for the M-acac when compared to the theoretical free ion value for *n*? Is the acac anion classified as a weak field or strong field ligand based on the value for n? (Support your reasoning.)

A third study is the utilization of the mass spectrum and modern 1D and 2D NMR experiments (^{1}H and ^{13}C , COSY, HSQC, HMBC, DQF-COSY, etc.) to identify an unknown disubstituted benzene. In addition to describing the merit of each NMR experiment to support your identification of the unknown, we are also expecting that you will predict carbon chemical shifts for the possible isomers and also decipher as many proton coupling patterns as the data support for your unknown (using the HNMR peak table provided in packet). You are expected to discuss the significance for each NMR experiment in the packet as it relates to identifying your unknown.

We look forward to reading what you have to say about the scientific analyses and the significance of your results.

Emile
John
& Chris