Chemistry 252 Experiment 64 Spring 2009

Introduction

The purpose of this experiment is to complete a project, described in Experiment 64, working in groups of 4. This is a two step synthesis involving an Aldol reaction to form a Chalcone followed by a Michael Addition of ethyl acetoacetate . Read the introduction to Experiment 64 (pages 533 and 534of PLKE).

There are 4 aldehydes listed on page 534 that you can use as the starting material in this experiment. The goal is for your group to complete a successful synthesis of the Michael/Aldol product from **two** of the aldehydes. It is up to each group to plan how to proceed with this project. In making plans, you should assign tasks so that **everyone** is doing productive work all three days. For example, when working on the first step (Experiment 41), you might start by having each person performing the synthesis with a different aldehyde.

You should now read the **Procedure** (pages 534 and 535) carefully. There is a lot of good information in this material. On page 535, at the end of the 2nd complete paragraph, Experiment 41 should be Experiment 42. See page 3 of this handout for a summary of the reactions in Experiment 41 and 42.

We will work on this experiment May 27, June 1 and June 3rd. You may do MP and spectra on June 8.

For the Pre-lab on Day 1 your group should write out the reaction equation for all 4 of the Chalcones **and** the reaction equation of for the final Michael /Aldol product for each of the 4 chalcones. Each group only needs to turn in one prelab.

You **do not** need to write a reaction table or any procedures.

Specific comments on Experiment 41:

Note that p-anisaldehyde is the same as 4-methoxybenzaldehyde. You should scale this procedure up (perhaps 3x or 4x) in order to have enough of the chalcone to do Experiment 42. The actual amount of p-anisaldehyde and piperonaldehyde required for this procedure is given in the book. If you start with one of the other aldehydes, you will need to calculate how much to use. The number of moles of the aldehyde should be the same in each case. The most challenging part of this experiment is usually getting the product to solidify from the reaction mixture. Several hints are given in the procedure and Footnote 2 on page 341.

Since the melting points for the chalcones from all 4 of these aldehydes are given, this may be sufficient to check the purity. You can also run a NMR or IR spectrum. **A high level of purity of the chalcone is essential before going on to Experiment 42**. Most likely you'll need to crystallize the crude product. A specific procedure is given in the book for crystallizing two of the products; however, you can crystallize all of them in the normal way: add a minimum amount of hot solvent required to dissolve the solid and then cool. 95% ethanol is usually a good solvent for these compounds.

Specific comments on Experiment 42:

To determine how much of your chalcone to use, you will need to keep the moles the same as the number of moles of *trans*-chalcone given in the procedure. **If you scale up or scale down the procedure, all amounts must be scaled up or down proportionately.**

ALERT!!!! There is one part of the procedure that may need to be modified. In the section on page 346 **Removal of Catalyst,** acetone is used to remove the product from the catalyst. Before adding acetone, make note of how much solid you have. The instructions call for 1.5 mL of acetone, but you may need to add more. Most of the solid should dissolve in the acetone. If this does not happen after adding 1.5 mL of acetone, you will need to add more acetone until most of the solid has dissolved. Don't add a lot more, but pay attention to the amount of solid and when no more seems to be dissolving, you can stop adding acetone.

Melting points can be used to check the purity of the final product in this part of the experiment. NMR and IR spectra can also be helpful. You can compare the IR to the one on page 347, even though your compound will be slightly different. The NMR data at the bottom of page 347 may also be helpful, but you may need some help to fully interpret the NMR. You should turn in **two products** with your name, name of aldehyde, weight, and mp.

I expect you to do this work without a lot of assistance from me. You should first try to work out any questions or problems within your group. If you still need help, then ask your instructor.

Lab Report and Grading

All notes taken in lab must go into your notebook.

Each group will hand in one lab report. Guidelines will be coming.

There will be a small subjective grade assigned individually to each student. It will be based on: how well you make use of lab time, how much you contribute to the project, how well you work together as a team, how well you seem to understand the work you are doing, your attitude towards this work, working safely, use of appropriate equipment, and use of notebook.

Each group will submit one lab report, consisting of the following items:

I. Introduction

This should be written in a style similar to the introductions to experiments in your textbook. In fact, it may look somewhat similar to the one in Experiment 64! You should discuss the reactions and possibly their mechanisms. You should include any other general information that you feel is important for your readers to know about. This should be written at a level appropriate for 2nd or 3rd quarter organic chemistry students.

II. Procedure

Write a detailed procedure in the style of PLKE. You may even use some of the phrases in the textbook without getting dinged for plagiarism! This should be the procedure that worked best. (Note: Do not write this up as a report of what you did. Instead you should write a procedure with instructions for someone else to follow.)

III. Experimental Results

In this section, you will present the results of all the syntheses (or experiments) that you performed, including the unsuccessful ones. You can write this in any form you think appropriate, but it should be clear and complete. This might be presented in the form of a table, where you give information about all the variables and the results used to monitor the success of each experiment. In addition to looking at clarity and completeness, I will be grading this for how reasonable and logical your approach was in trying to find a workable procedure.

IV. Conclusion

Discuss the experimental results. Are there problems in the procedure that still need work? What do you suggest should be done next if more work is required? Include anything else you feel is important.

Subjective Grade

This will be assigned individually to each student. It will be based on: how well you make use of lab time, how much you contribute to the project, how well you work together as a team, how well you seem to understand the work you are doing, your attitude towards this work, working safely, use of appropriate equipment, and use of notebook.