Michael and Aldol condensation reactions (PLKE #64)

This 2 step synthesis starts with PLKE 37 (Aldol condensation) followed by PLKE 38 (Michael addition/cyclization). This synthesis is summarized in PLKE #64 pg 564. You can choose which Michael/Aldol product to make starting from either 4-chlorobenzaldehyde, 4-methoxybenzaldehyde, 4-methylbenzaldehyde or piperonaldehyde (your choice).

Specific comments on Experiment 37: 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 38. 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 339.

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. A high level of purity of the chalcone is essential before going on to Experiment 38. 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 38: 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 344 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.

You should turn in your product with your name, name of aldehyde, weight, and mp.

To Prepare for this lab:

Read section 19.11, 19.13 (Aldol condensation) and 19.17 of Bruice and PLKE #64 for a review of the reactions.

Prelab to be turned into your instructor before you begin:

1) Write the balanced reaction equation for each step of the synthesis using the aldehyde you chose.
REPORT GUIDE Report Due: checkout

This will not be a formal report. This report will be done individually.

NAME_____________________________

Results:

Turn in your final Michael/Aldol product that you produced. Label the vial with your name, structure and weight and mp of product.

Attach the NMR spectra of the final Michael Aldol product you produced with proper NMR interpretation.

Draw the structures the chalcone and Michael/Aldol product that was attempted. By each structure record the weight & mp

Show both the mechanism for the chalcone formation as well as the Michael/Aldol product formation (you can use the back of this page if needed).