

CHEMISTRY 344 - Organic Chemistry Laboratory II – Spring 2012
Lab #6: Green Chemistry Reaction Enigma

In this lab, you will prepare a reaction mixture consisting of three solid reactants, that you will grind together in the absence of any solvent. Your goal is to isolate, purify, and identify your product based on your knowledge of organic reactions, and the product melting point and IR spectrum.

Pre-lab Reading: You are expected to read the experimental procedure in this handout. The IR and NMR spectra of the organic reactants, **3,4-dimethoxybenzaldehyde** and **1-indanone**, can be found at the end of this handout. You should also review all reactions you have seen this semester. Based on the reaction conditions, you should be able to easily identify the organic reaction and product.

Pre-Lab Notebook Preparation: Complete the following information in your lab notebook before your pre-lab recitation. Use the numerals and headings as given.

Table of Contents: You will need to update the Table of Contents for each new lab with the date and brief title of each experiment, and the notebook page number on which the lab begins.

I. Title of the Experiment and Date.

II. Purpose of the experiment: One- or two-sentences describing what you are going to do and why. State your experimental goals.

III. Equations: Write the balanced equation for this reaction, *including structures*.

IV. Table of Reagents: In your lab notebook create a table, like the one shown below, for the following reagents used in this lab: **3,4-dimethoxybenzaldehyde** and **1-indanone**. Your table must include the name, structure, and all physical data. You can find this information in the Aldrich Chemical Catalog, the Merck Index, or on-line from the **ChemIndex** web site. The link to ChemIndex and instructions can be found on Dr. Brush's Lab web site: <http://webhost.bridgew.edu/ebrush/CH343%20Lab.htm>.

Chemical name and formula	structure	molecular mass (g/mole)	melting point (solids) (°C)	boiling point (liquids) (°C)	density (liquids) (g/mL)
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V. Procedure: Summarize the lab procedure in your notebook. Do not copy the procedure word-for-word.

VI. Health, Environmental, and Waste disposal: Look up and summarize some of the health and environmental hazards for **3,4-dimethoxybenzaldehyde** and **1-indanone** at the MSDS database:

<http://hazard.com/msds/index.php>

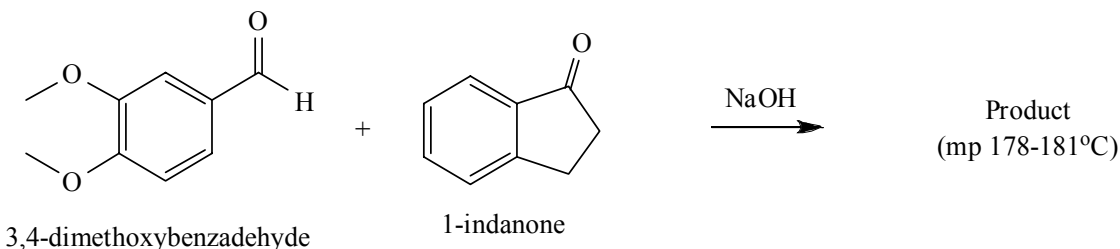
Laboratory Book: The following should be completed before leaving lab, initialed and dated by you.

VII. Data and Observations: Record a rough transcript of your experimental method in your lab notebook, indicating what you actually did and what you actually observed. Do not prepare this section in advance. This section should be written in a paragraph format and include: **experimental procedure, all reagent mass and volume measurements, observations, crude and pure product mass or volume, % yield calculations, and product analysis by melting point, chromatography analysis, or instrument analysis.** You will be judged on the depth of your observations and technical success (yields, purity, etc.).

VIII. Discussion and Conclusions: A brief, but critical evaluation of your results, and the success of your experiment. Address the experiments goals and purpose!

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Experimental Procedure



(1) Place 0.25 g of 3,4-dimethoxybenzaldehyde and 0.20 g of 1-indanone in a screw cap centrifuge tube. Using a metal spatula scrape and crush the two solids together until they become a brown oil. Be careful that you don't break the test tube!

(2) Add 0.05 g (covers half of your small spatula) of finely ground solid NaOH to the reaction mixture and continue scraping until the mixture becomes a solid. This will take approximately 10 min.

(3) Allow the mixture to stand for 15 minutes, then add about 2 mL of 10% aqueous HCl solution. Mix well with your spatula and be sure to scrape down any product from the sides of the tube. Check the pH of the solution to make sure it is acidic.

(4) Isolate the solid by vacuum filtration on a Hirsch funnel. Use cold water to rinse your tube, if necessary. Pull air through the crude product for 2-3 minutes to partially dry the solid. Determine the mass of the crude product.

(5) Obtain about 20 mL of the recrystallizing solvent, 90% ethanol / 10% water. Transfer your crude product to a 25 or 50 mL vial, beaker or flask, and add a magnetic stir bar. Add those 20 mL of the solvent, and heat the mixture on a hot plate with vigorous stirring until the solid dissolves giving a brownish solution. You should heat the mixture to just below its boiling point. You should not require more than 20 mL of solvent to effect crystallization.

(6) After all the crude product has dissolved, remove the flask from the hot plate, and allow it to cool slowly on your lab bench for 5-10 minutes. Once recrystallized product begins to appear, place the flask on ice for 5 minutes, then isolate the product by filtration on a Hirsch funnel, rinsing the flask with ice-cold water. Dry for 5 minutes, then scrape onto a clean watch glass and dry in an oven for 10-15 minutes at 115°C.

*NOTE: If product crystals do not appear upon cooling, you may need to scratch the inside of the flask with a glass stirring rod.

(7) Cool your product to room temperature, and determine the mass of recrystallized product, its melting point, and obtain its IR spectrum. The NMR and IR spectra of the organic reactants are attached at the end of this handout.

Name: _____ Lab Partner _____

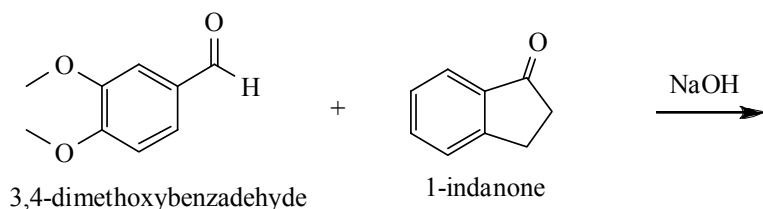
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Lab Report Grade _____ (100 points)

The following questions are based on your pre-lab reading and the results of your experiment. You and your lab partner are encouraged to work together, but you must each turn in your own report! Answer each question in the space provided, and **show all calculation work below or on a separate sheet of paper.**

1) What reaction is this? Explain how you know.

2) Complete the reaction below by drawing the structure of the expected product and balancing the equation.



3) Mass of 3,4-dimethoxybenzaldehyde used _____ (g)

4) moles of 3,4-dimethoxybenzaldehyde used _____ (moles)

5) Mass of 1-indanone used _____ (g)

6) moles of 1-indanone used _____ (moles)

7) Limiting reactant _____

8) Theoretical yield of product _____ (g) _____ (moles)

9) Experimental yield of crude product _____ (g) _____ (moles)

10) Percent yield of crude product _____

11) Experimental yield of recrystallized product _____ (g) _____ (moles)

12) Percent yield of recrystallized product _____

13) Observed mp of recrystallized product _____ (lit 178-181°C)

14) Based on the % yield of your recrystallized product, and its melting point, discuss the efficiency of this reaction. A paragraph of several sentences is expected.

15) The IR spectra for the organic reactants are attached at the end of this handout. Do you expect that the IR spectrum of your product will be significantly different than either organic reactant? Briefly Explain.

16) The ^1H NMR spectra for the organic reactants are attached at the end of this handout.

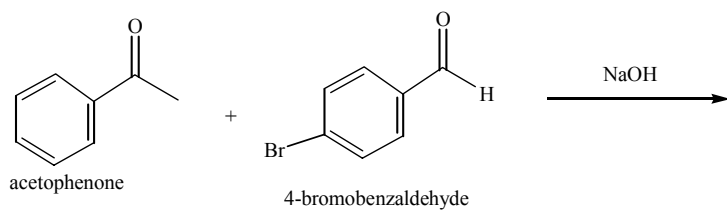
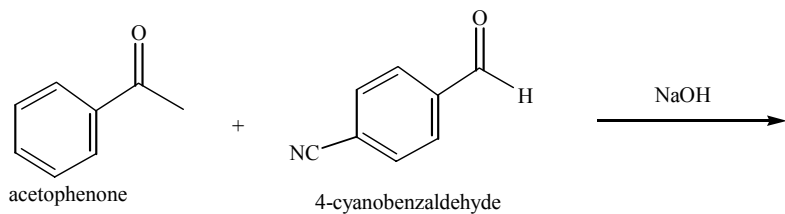
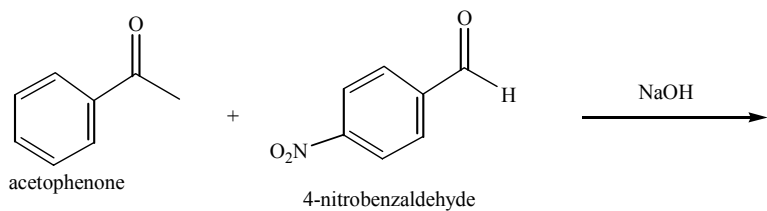
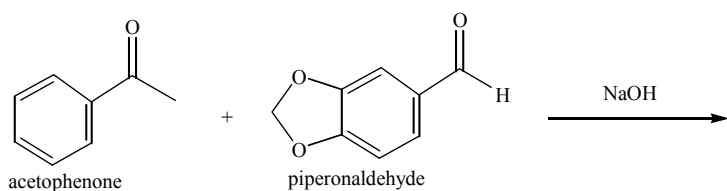
(a) What proton signal from the NMR of 3,4-dimethoxybenzaldehyde do you predict will not be seen in the NMR of your product?

(b) What proton signal from the NMR of 1-indanone do you predict will be different in the NMR of your product?

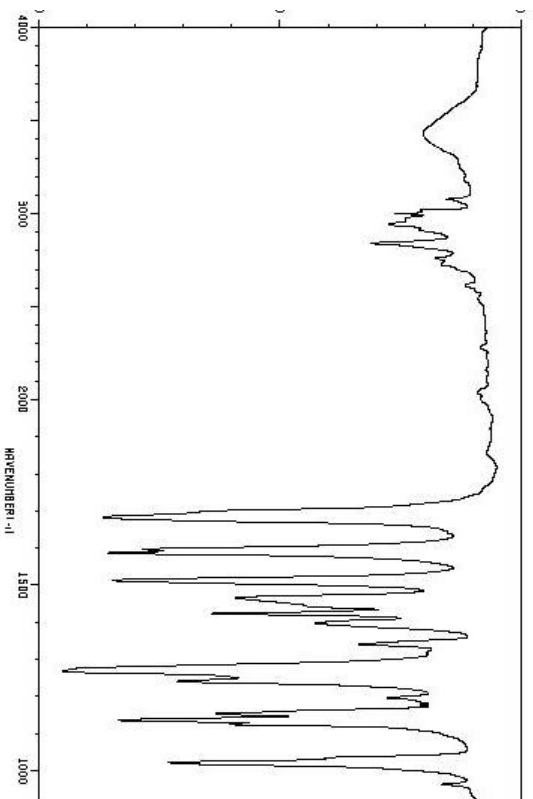
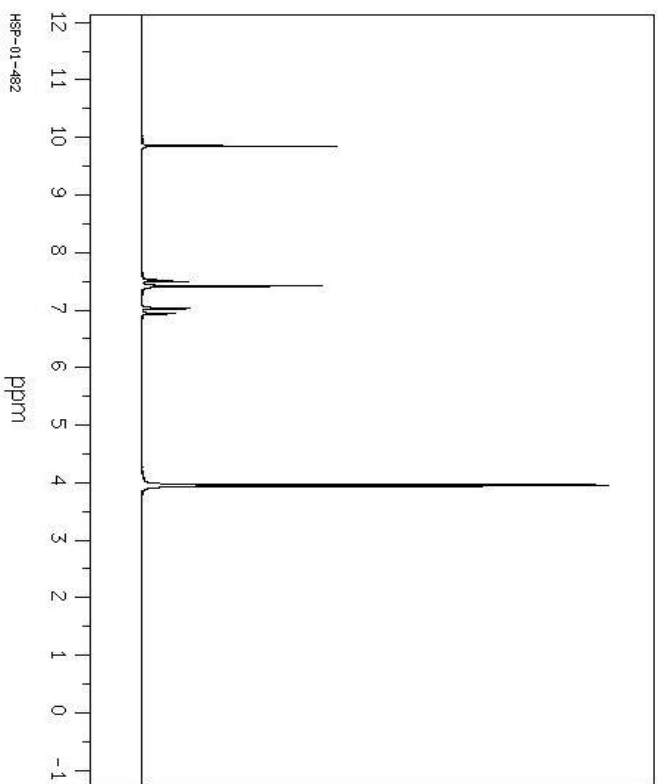
(c) What proton signal of your product is unique as compared to both reactants?

17) This reaction was done in the absence of solvent. In your own experience over the past year in the organic chemistry lab, is this “greener” than a typical reaction? Briefly explain.

18) Based on your knowledge of this reaction, draw the structure of the organic product(s) for each of the following related reactions:



1H NMR and IR for 3,4-dimethoxybenzaldehyde



1H NMR and IR for 1-indanone

