Lab 13: Predicting the Products of an Aldol Reaction

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Objectives

By the end of this laboratory, you should have developed the skills to do the following:

- Select an appropriate recrystallization solvent.
- Predict and identify the product of a reaction.

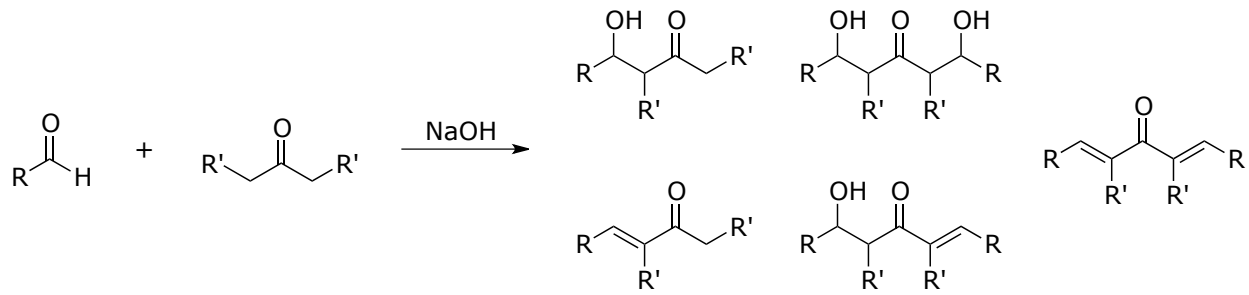
Recommended Resources

- Website ~ Aldol Addition on the Organic Chemistry Portal
  http://www.organic-chemistry.org/namedreactions/aldol-addition.shtm
- Website ~ Aldol Condensation on the Organic Chemistry Portal
  http://www.organic-chemistry.org/namedreactions/aldol-condensation.shtm
- Tutorial ~ Introduction to Crystallization Tutorial
  https://www.youtube.com/playlist?list=PLC37691582723F6F2

Background

In this experiment you will react an aldehyde with a ketone under basic conditions. You will use your knowledge of organic chemistry and reaction mechanisms to predict the possible reaction products.

Under basic conditions, it is expected that the aldehyde and ketone will react to form an aldol addition product. In some situations, further reaction may lead to formation of the aldol condensation product. Additionally, consider that we are using more than 2 molar equivalents of the aldehyde, which may result in further reaction of the aldehyde with the ketone. The five possible products are shown below.



Since everyone is using a different aldehyde and ketone, reaction temperatures and times will vary significantly. Unless it is very obvious that your reaction is complete (as indicated by your product crystallizing out of the reaction solution), you will have to monitor your reaction by TLC to determine when it has finished. In an ideal situation you can tell that a reaction is complete based on the disappearance of the limiting reagent. However, if your limiting reagent is not UV active, you will need to look for the appearance of a new product spot. Your reaction is complete when the product spot is the major spot or is no longer increasing in size.

After the reaction is complete, you will need to purify it via recrystallization. As each product is different, you will have to find an appropriate recrystallization solvent to purify your product. It is recommended that you test each of the three suggested solvents (95% ethanol, toluene, or 2-butanone) to see which works best for recrystallizing your particular product. You can then determine the identity of your product using melting point, IR, and NMR.

You will be assigned an aldehyde and a ketone from the table below. Prior to coming to lab, you will need to calculate the grams/volume needed for your particular set of reagents.

	Aldehydes
A	benzaldehyde
B	4-methylbenzaldehyde
C	4-methoxybenzaldehyde
D	*trans*-cinnamaldehyde

	Ketones
I	acetone
II	cyclopentanone
III	cyclohexanone
IV	4-methylcyclohexanone

Lab Notebook Preparation

Before coming to lab, the following items must be in your lab notebook:

1. Title of experiment
2. Date the experiment is to be performed
3. The chemical reaction you are attempting (with skeletal structures of the specific aldehyde and ketone you are using)
4. A table with information about your starting materials. Include molecular weight, molar equivalents, and mmoles to be used. For solids include grams. For liquids, include grams, density, and volume. For solutions, include the concentration and volume.
5. The molecular weight and theoretical yield of any possible products
6. Any relevant physical properties (i.e., boiling/melting points of the starting materials and expected products, if known)
7. Hazards of and appropriate precautions for the safe handling of your particular aldehyde and ketone
8. References

Safety Notes

- Assume that all aldehydes and ketones are flammable and harmful by inhalation, ingestion, and skin absorption. Do not inhale their vapors and avoid contact with eyes, skin and clothing.

Directions

1. Accurately measure 1.5 mmol of the ketone you have been assigned into a flask or vial and dissolve it in 3 mL of ethanol. (Use your smallest round bottom flask or the microscale kits.)
2. Stir in 6.0 mmol of the aldehyde you have been assigned.
3. Add 2.5 mL of 1.0 M NaOH.
4. Let the reaction mixture stir at room temperature for 10 minutes. If you see a significant amount of crystal formation, your reaction is complete and you can skip to step 7.
5. TLC your reaction to determine if it is complete. Be sure to dilute your aldehyde before spotting it on the TLC plate. The ketones are not UV active so it is not necessary to include them on the TLC. You will have to try different solvent systems to find one that separates the components of your reaction enough to tell what is going on. (It is recommended that you start with a mixture of 25% ethyl acetate in hexane and then adjust as needed.)
6. If your reaction is not complete after the 10 minutes, heat your reaction under reflux until it is complete (or nearly so) as indicated by TLC.
7. Once your reaction is done, allow it to cool to room temperature, and then cool it further in an ice-water bath.
8. Collect the product by vacuum filtration, wash it with approximately 1-2 mL of cold 4% (v/v) acetic acid in ethanol, and then wash with approximately 1-2 mL of cold ethanol.
9. Recrystallize the product using 95% ethanol, toluene, or 2-butanone. (Note: You will have to test these solvents to determine which will work best for recrystallizing your product. Use about 100 mg in 1 mL of solvent as a test.)
10. Dry and weigh your product.
11. Determine the identity and purity of your product using melting point, IR, and ^{1}H NMR. (Note: If you only have enough product for one experiment, do ^{1}H NMR.)
12. If needed, obtain a ^{13}C NMR.

Reporting Your Results

Write your report according to the guidelines described in "Topic 4: Writing an Organic Chemistry Lab Report". Work by yourself on this report.

References & Additional Resources

1. Lehman, J. W. *Operational Organic Chemistry: A Problem-Solving Approach to the Laboratory Course*, 3rd ed.; Prentice Hall: Upper Saddle River, NJ, 1999; pp 513-514.