

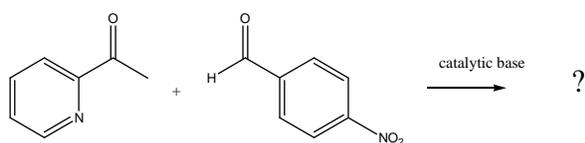
Effect of Reaction Conditions on the Aldol Reaction

S343

Question: How do temperature and choice of base (reaction conditions) affect the outcome of an aldol reaction?

Background

The aldol reaction is a very important means of making carbon-carbon bonds. Under basic conditions, the α protons of ketones, aldehydes, and esters may be removed to form strong nucleophiles that can then add to another compound containing a carbonyl. While this can lead to a mixture of products, there are ways to favor formation of one product. For example, if a ketone is mixed with an aldehyde containing no α protons, there is only one possible nucleophile. Furthermore, the aldehyde is a much better electrophile than a ketone under these conditions, so the aldehyde serves as the electrophile preferentially. An example of such a “crossed Aldol” reaction is shown below:



The outcome of any reaction depends on the reaction conditions. Choices of solvents, temperatures, and bases can lead to an effective or ineffective reaction. In some cases, these changes can even lead to different products being formed. For example, in the reaction above, there is the possibility that the product formed will be the aldol addition product; it is also possible that the reaction will continue on to form the aldol condensation product.

Your team will investigate two parameters: temperature and choice of base. You should run the reaction at room temperature and 60 °C, and you should compare the reaction using sodium carbonate to the reaction with sodium hydroxide. Based on your knowledge of the aldol reaction, you should have a prediction of the expected product or products. After the reaction is complete, you should characterize the resulting compound to determine its actual identity.

Procedures:

In a 25 mL roundbottom flask equipped with a spin bar, add 0.0010 mole (___g, ___mL; density = 1.08 g/mL) of 2-acetylpyridine and 10 mL of water. (Always work with 2-acetylpyridine in the hood. Strong odor—do you recognize it?) Stir the mixture vigorously. In a separate vial, dissolve 0.0010 mole (___g) nitrobenzaldehyde in a minimum amount of hot methanol. While still hot, add the solution of aldehyde into the vigorously stirring mixture in the roundbottom flask. Add 0.00025 mol (___g) of base (either sodium carbonate or sodium hydroxide) dissolved

in 5 mL of water. Stir for one hour. (If heating, attach a reflux condenser to the reaction flask and heat in a hot water bath. Do not allow the water bath to exceed 65 °C.) Cool the reaction in an icebath and collect the precipitate by suction filtration. Wash the solid with water (3x 10mL.) Dry as thoroughly as possible before obtaining characterization data.

Results:

Include all characterization data you obtained to determine the structure, purity, and yield of each product.

Comments:

What product was isolated for your set of reaction conditions (or was only starting material isolated?) What data support this conclusion? Ask other students who used different conditions about their conclusions.

How did the choice of temperature and base affect the outcome of the reaction? Give a rational explanation of these outcomes.

Calculate a theoretical yield of your product and determine the % yield of your reaction.

Conclusions: The outcome of this aldol reaction is/is not affected by changing temperature and base.

Lab 10 assignment: Turn in a hardcopy of your lab notebook (carbon copy or photocopy)

- Due at the beginning of lab next week
- 25 pts based on in-lab performance, completion of all sections, correctness of content
- No electronic submission to turnitin.com