ALDOL CONDENSATION

OVERVIEW: This lab not only illustrates the aldol condensation reaction, but also introduces small scale preparation methods.

PRE-LAB: A table like the following must be completed in your lab notebook before coming to lab.

To calculate the theoretical yield, you must first determine which reactant is the limiting reagent and then base your calculations on this compound. See Appendix D, *Theoretical and Percent Yield* for more information.

<table>
<thead>
<tr>
<th>benzaldehyde</th>
<th>+</th>
<th>acetone</th>
<th>-</th>
<th>dibenzal-acetone</th>
<th>+</th>
<th>other products</th>
</tr>
</thead>
<tbody>
<tr>
<td>structural formula</td>
<td></td>
<td>structural formula</td>
<td></td>
<td>structural formula</td>
<td></td>
<td></td>
</tr>
<tr>
<td>molar mass</td>
<td></td>
<td>molar mass</td>
<td></td>
<td>molar mass</td>
<td></td>
<td></td>
</tr>
<tr>
<td>starting grams</td>
<td></td>
<td>starting grams</td>
<td></td>
<td>theo. yield in grams</td>
<td></td>
<td></td>
</tr>
<tr>
<td>starting moles</td>
<td></td>
<td>starting moles</td>
<td></td>
<td>theo. yield in moles</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

BACKGROUND: The term "aldol condensation" applies to a variety of well-known reactions involving aldehydes, ketones, and esters.

The carbon next to the carbonyl (C=O) carbon of an aldehyde or ketone is called the \( \alpha \)-carbon. The carbon next to the \( \alpha \)-carbon is called the \( \beta \)-carbon. The \( \alpha \)-carbon is acidic because of the stabilizing effect of the carbonyl group. This means it will easily lose a hydrogen atom in a basic solution.

In a typical aldol condensation, a strong base pulls off a hydrogen atom on the \( \alpha \)-carbon, to produce a negative charge. This negatively charged carbon then attacks the slightly positive carbon of the carbonyl group on another molecule, giving a \( \beta \)-hydroxy carbonyl compound. If the reactant is an aldehyde, this product is an \textit{aldehyde-alcohol}, or \textit{aldol} product. The same term is also used for similar products from ketones.
Here is an example of the reaction, using acetaldehyde:

\[
\begin{align*}
\text{CH}_3\text{CHO} & \quad \text{OH}^- \\
\text{CH}_2\text{CHO} & \quad \text{CH}_2\text{CHO} \\
\end{align*}
\]

If the product from the aldol condensation has a benzene ring attached to the \( \beta \) carbon, then the product will usually spontaneously dehydrate to give an \( \alpha, \beta \) unsaturated ketone or aldehyde. Also, if a ketone is used that has \( \text{H} \) on both \( \alpha \)-carbons, attack will occur on both ends of the ketone, giving an even more complex product. Today's lab illustrates these points in the reaction of benzaldehyde and acetone. Consult your text for more information on this type of reaction and its mechanism.

\[
\begin{align*}
\text{2 Benzaldehyde} + \text{CH}_3\text{CCH}_3 & \quad \text{OH}^- \\
\text{Dibenzalacetone} & \quad \text{Dibenzalacetone} \\
\end{align*}
\]

**SAFETY:** Benzaldehyde and acetone are highly flammable. Sodium hydroxide is corrosive. Apron, goggles, and gloves must be worn during this experiment.

**PROCEDURE:** Into a small test tube that will fit in a centrifuge, add as accurately as you can (from a 50 mL buret located in the side hood) 1.5 mL of Solution A (this is a mixture of compounds in the proportions: 10g NaOH, 100 mL water, and 80 mL ethyl alcohol).

Into the same tube, add from a 10 mL buret in the side hood 0.1 mL of Solution B (this is a mixture of compounds in the proportions: 50.0 mL benzaldehyde and 18.0 mL acetone).
While holding the test tube firmly, carefully tap it with your finger to mix the reagents. Continue to tap the tube occasionally for the next 15 minutes and note the precipitation of the yellow dibenzalacetone.

Place the tube in a centrifuge. Balance this tube by placing opposite it another tube with an equal amount of water. Centrifuge the tube 1-2 minutes and remove the supernatant liquid using a pipette or dropper.

The product needs to be recrystallized, but such a small amount requires a slightly different technique from the one you've used before. To recrystallize the product, warm the tube in a hot water bath and add about 0.5 mL of ethyl acetate to dissolve the product. Remove the water remaining as a layer under the ethyl acetate with a pipette. Let the tube cool, then place in an ice bath. Tap it occasionally to induce crystallization. (If no crystals form, return the tube to the water bath and heat it to drive off some of the solvent.) When crystallization is complete, collect the product by suction filtration and rinse it with a little ice cold methanol. Dry, weigh, and determine the melting point.

PRODUCT CHARACTERIZATION: The product has double bonds. Double bonds are oxidized in a reaction with KMnO$_4$. In your notebook, show the reaction that takes place between KMnO$_4$ and an alkene.

Confirm the presence of double bonds by dissolving a small amount of the product in methanol in a test tube. Add two drops of the 1% KMnO$_4$ test solution. If a double bond is present, the purple color will immediately disappear, replaced by a brown precipitate.

DISPOSAL: A waste jar is provided in the side hood for disposal of the product. Another jar is provided for the waste product of the KMnO$_4$ reaction.

CALCULATIONS: Since you mixed solutions together, the calculation of yield is a little more complex than usual. In order to determine the theoretical yield, you must first determine the moles of each reactant you started with. The reactants came from a solution that was in the ratio of 50 mL benzaldehyde and 18 mL of acetone in a total of 68 mL. Therefore, the fraction of benzaldehyde is 50/68 and the fraction of acetone is 18/68. Since you used 0.1 mL of the solution, the volume of benzaldehyde used is actually 0.1 mL x 50/68 and the volume of acetone is 0.1 x 18/68 mL. Using the density of benzaldehyde and the density of acetone, you can
calculate the starting grams of each organic reactant. From this you can calculate the starting moles.

**LAB NOTEBOOK:** Use tables to summarize the results of the reaction.

<table>
<thead>
<tr>
<th>theoretical yield (grams)</th>
<th>actual yield (grams)</th>
<th>percent yield</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>product</th>
<th>observed mp</th>
<th>literature mp</th>
</tr>
</thead>
<tbody>
<tr>
<td>dibenzal-acetone</td>
<td>[IUPAC name]</td>
<td></td>
</tr>
</tbody>
</table>