REACTION: Aldol Condensation

In this experiment, you will prepare an aldol condensation product using an unknown aldehyde and an unknown ketone. You will then identify the aldehyde and ketone by analyzing the $^1$H NMR and the melting point of your condensation product and then backtracking to your starting materials.

You will be assigned an aldehyde and a ketone from the possibilities below. Note that all aldehydes are aromatic aldehydes and, therefore, carry no $\alpha$-hydrogens and all ketones are non-aromatic.

**Ketones:**

- acetone
- cyclopentanone
- cyclohexanone
- 4-methylcyclohexanone

**Aldehydes:**

- $p$-tolualdehyde
- $p$-anisaldehyde
- cinnamaldehyde
Melting Points of the Possible Aldol Condensation Products

<table>
<thead>
<tr>
<th>Aldehydes</th>
<th>Ketones</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Acetone</td>
</tr>
<tr>
<td>p-Tolualdehyde</td>
<td>175 °C</td>
</tr>
<tr>
<td>p-Anisaldehyde</td>
<td>130 °C</td>
</tr>
<tr>
<td>Cinnamaldehyde</td>
<td>144 °C</td>
</tr>
</tbody>
</table>

READING ASSIGNMENT:
- This handout for procedure
- Supplementary information in Janice Gorzynski Smith (2nd Ed.) Chapter 24.

PRE-LAB ASSIGNMENT:
- Complete all portions of pre-lab notebook work according to the guidelines.
- Rewrite the procedure in your lab notebook using your own words.
- Sapling assignment.

CAUTION
The aldehydes and ketones used in this experiment are skin and eye irritants. Do all work in the hood, wear gloves, and avoid all contact with skin, eyes, and clothing. Aqueous sodium hydroxide solutions are corrosive and cause burns. Wear gloves, and avoid all contact with skin, eyes, and clothing.

EXPERIMENTAL:

For this experiment, you will receive two vials filled with liquid samples: one is an aldehyde and the other is a ketone. First, identify which vial contains aldehyde (or ketone) by taking the IR spectrum of ONE sample. One IR spectrum is good enough to identify which vial contains aldehyde or ketone. **Hint:** Where do you find the characteristic aldehyde C-H absorption bands? See Table 20.2, in *Techniques*. A ketone should have no characteristic aldehyde C-H absorption bands.

After identifying which vial contains the aldehyde, measure out 0.80 mL of it and place it in a 25 mL Erlenmeyer flask. Measure 0.20 mL of the ketone and add it to the flask containing the aldehyde. Add 4.0 mL of 95% ethanol and 3.0 mL of 2.0 M NaOH to the flask and stir until no more precipitate forms. If precipitate formation is slow at room temperature (taking longer than 15 min), heat the reaction using a boiling water bath. The condensation products are pretty colorful! What governs the color of products?

Once the reaction is judged complete and no more precipitate is forming, cool the reaction to room temperature, if you were heating it, and then place it in an ice bath for 10 min. While you are waiting for the reaction to cool in your ice bath, measure 8 mL of 95% ethanol and 4 mL of 4% acetic acid/ethanol solution in separate test tubes or Erlenmeyer flasks and place those in the ice bath.
Use vacuum filtration to collect the product from the reaction mixture, rinse with 4 mL of the chilled ethanol, followed by the chilled acetic acid/ethanol solution, and finally the remaining 4 mL of chilled ethanol. Allow your products to dry under vacuum for 10 min while you clean up.

Obtain a melting point of your dry product. Your TA will select certain groups to prepare NMR samples. If your group is selected, prepare an NMR sample by dissolving ~100 mg of your product in 0.5 mL CDCl₃. If only a portion of the solid dissolves, filter the solution through a pipet filter before transferring it to the NMR tube.

Before going home, ensure you go to the stockroom to pick up a standard ¹H NMR of your product from the stockroom. You will need to know the unknown letters for both of your unknowns.

LAB WRITE-UP:

Write a complete lab report as described in the Report Guidelines for Students handout.

Here are some pointers on what to put in the Discussion Section, but are far from exhaustive. By this point, you should have an idea of what things should be talked about in the discussion section, regardless of what experiment you run:

- Determine the structure of your product. Clearly explain your reasoning for the assignments. Attach the NMR spectrum with your proposed product structure drawn on it at the end of your report. Determine the identity of the starting aldehyde and ketone from this information.
- Report the theoretical yield and percent yield of your product. Hint: You will need to use your conclusions from characterization data to calculate yield! At the end of your report attach a sheet showing all your work/calculations in order to receive full credit – it is not included in the 2 page limit.

In addition, draw out the full mechanism of condensation for the aldehyde and ketone you used in lab. This is not included in the two-page lab report but should be attached to it.