Minilab 34 – Preparation of Aldol Condensation Products

Goal
To perform a Claisen-Schmidt condensation reaction.

Reading and Working Ahead
Below is a list of aldehydes and ketones that will be used in this experiment. Perform the prescribed calculations for the two highlighted on your handout. These will be the chemicals you will use in your particular experiment. Calculate the mass of the ketone, to the hundredth place, and volume of the aldehyde, rounding up to the nearest milliliter.

<table>
<thead>
<tr>
<th>Ketones</th>
<th>Aldehydes</th>
</tr>
</thead>
<tbody>
<tr>
<td>acetone</td>
<td>benzaldehyde</td>
</tr>
<tr>
<td>cyclopentanone</td>
<td>4-methylbenzaldehyde</td>
</tr>
<tr>
<td>cyclohexanone</td>
<td>4-methoxybenzaldehyde (para-anisaldehyde)</td>
</tr>
<tr>
<td></td>
<td>trans-cinnamaldehyde</td>
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</tbody>
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Procedure
- Weigh the ketone into a test tube. First insert the test tube into a small graduated cylinder holder. Place this on the balance and tare. Be sure the balance reads to three places past the decimal. Add the ketone drop wise; 6-8 drops will probably be needed. Record the actual mass of the ketone you are using.
- Measure the aldehyde by volume, using a 1 mL volumetric pipet.
- No recrystallization – save solid until the next week to determine mass and melting point.

Pre-lab Questions
1. Show the mechanism and products (through dehydration) for the following Claisen-Schmidt condensations.
   (a) trans-cinnamaldehyde + 3-pentanone
   (b) benzaldehyde + cyclobutanone

2. Why do the reactions in the previous problem proceed through the dehydration step, spontaneously?

Follow-up Questions
1. What would have been the effect of not cooling the reaction flask prior to filtration?

2. What is the purpose of the acetic acid wash?

3. Why do you think it is possible to simply round the volume of the aldehyde up to the nearest milliliter?

4. Why didn’t we collect the crystals by gravity filtration?