Honors Cup Pre-Proposal: Synthesis of Cinnamaldehyde

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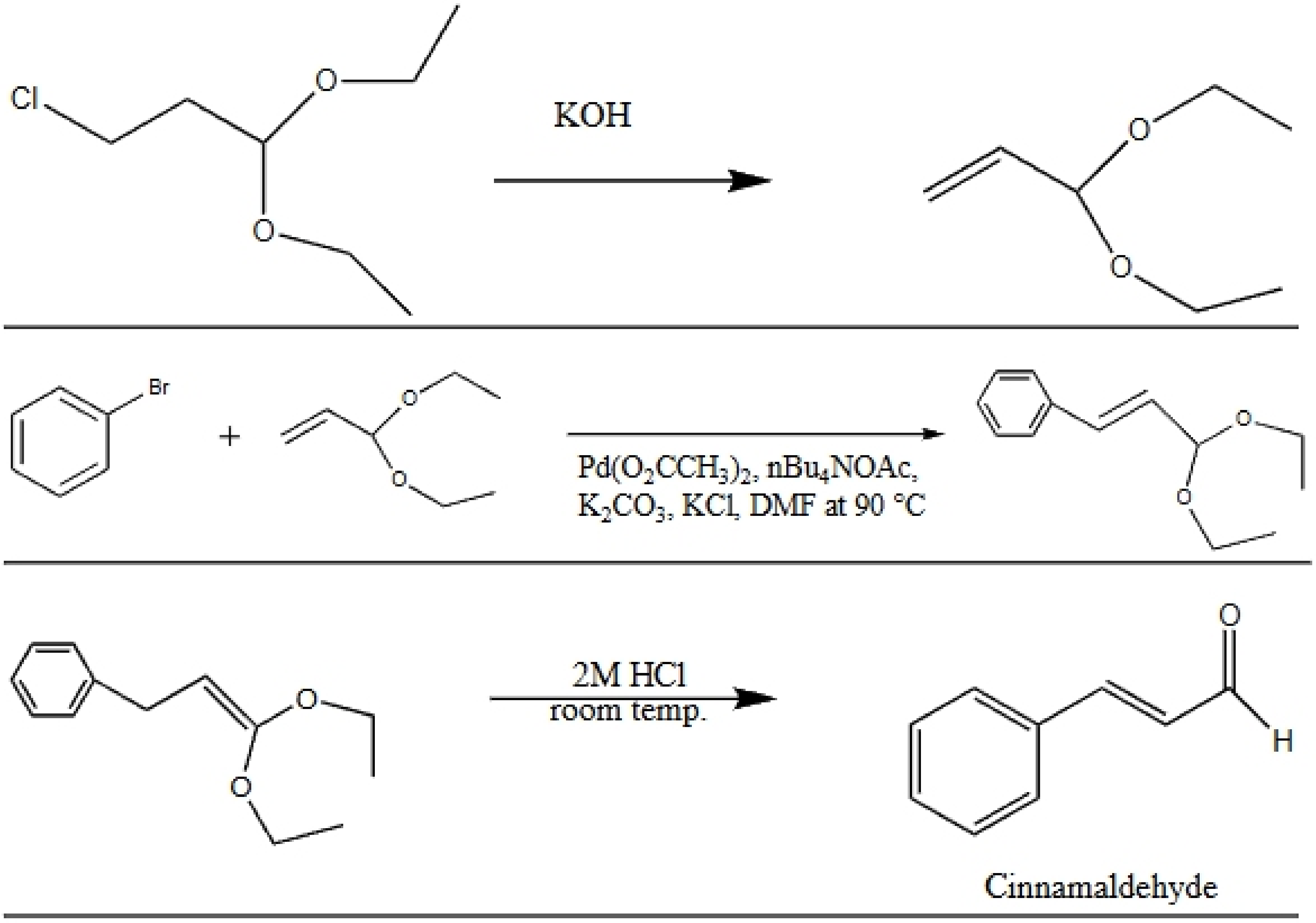
Group: 251-III

Introduction:

We will be attempting the synthesis of cinnamaldehyde, (E)-3-phenyl-2-propenal, which is the compound responsible for cinnamon's distinct odor and flavor. In addition to being used to flavor foods and in perfumes, cinnamaldehyde can be applied to the roots of plants as a low toxicity fungicide. Furthermore, this compound can be used as a corrosion inhibitor for ferrous alloys. It can be found naturally in the bark of cinnamon trees, camphor and cassia. Cinnamaldehyde is a mono-substituted benzene ring.

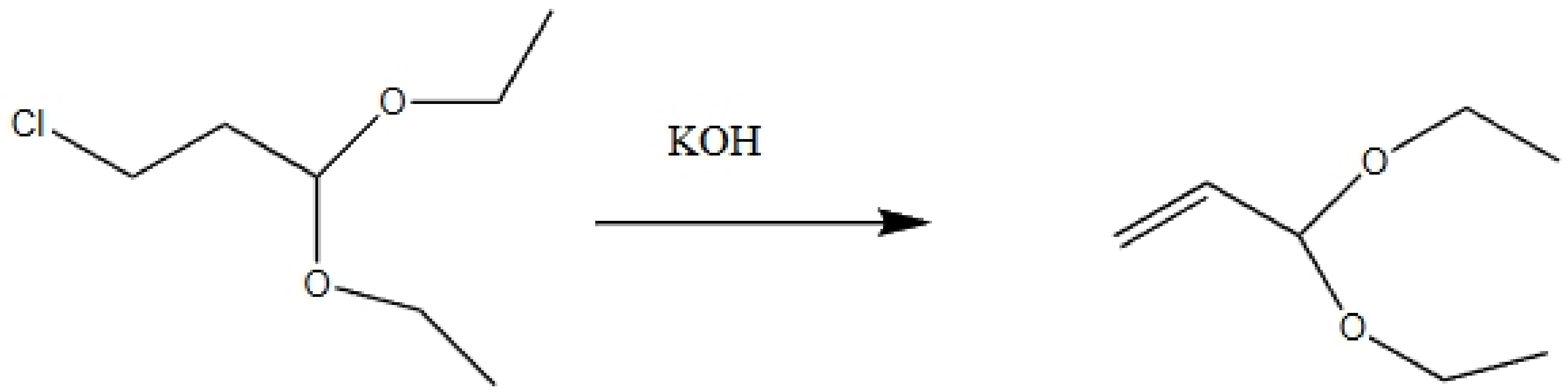
Reaction Scheme:



Step 1:

b-chloropropionaldehyde acetal

acrolein acetal

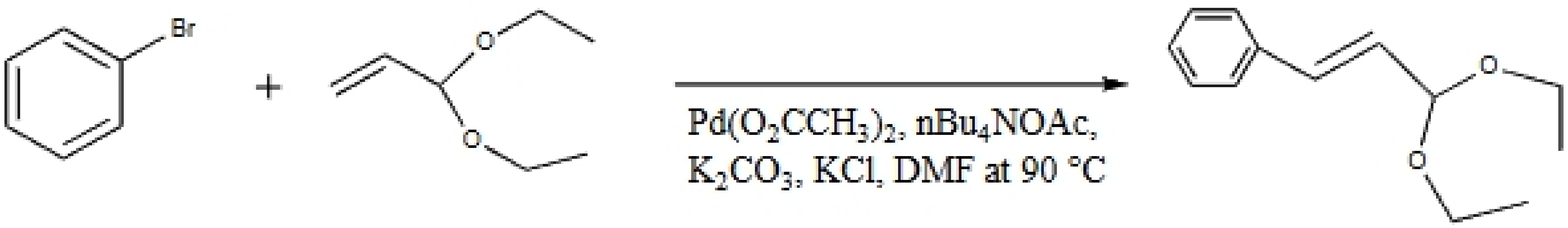


Combine dry, powdered KOH (20g) and b-chloropropionaldehyde acetal (10g) in 250 mL short neck round bottom. Shake the mixture vigorously and immediately attach to a three-bulbed Glinsky and set up for distillation. Heat the round bottom flask in an oil bath at 210 to 220 degrees Celsius for the distillation. The acrolein acetal will be the distillate (part that is distilled off). Place the distillate in a separatory funnel and remove the lower aqueous layer. Dry the organic layer containing the acrolein acetal over 10g of potassium carbonate; vacuum filter and distill (the product boils at 122 to 126 degrees Celsius).

Modifications: Use a Vigreux column instead of the Glinsky column.

Step 2:

bromobenzene acrolein acetal



Combine bromobenzene (78.5mg, 0.5 mmol) and acrolein acetal (180mg, 1.5 mmol) in a flask containing the following catalytic amounts:

DMF: 2mL

Palladium(II) acetate: 3.4mg, 0.015 mmol

Tetrabutylammonium acetate: 302mg, 1 mmol

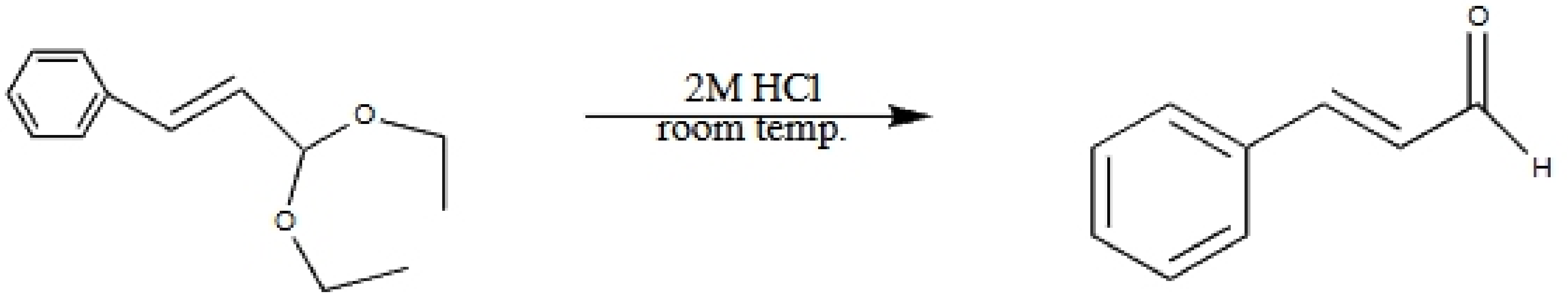
Potassium carbonate: 104mg, 0.75 mmol

Potassium chloride: 37mg, 0.5 mmol

Add stir bar to flask and allow mixture to stir for 1.5 hours at 90 degrees Celsius. After 1.5 hours TLC the reaction to make sure that all of the acrolein acetal has reacted. If some acrolein acetal remains, allow the reaction to continue and monitor with TLC until there is no acrolein acetal. After the reaction is complete, allow the product to cool down to room temperature before continuing on.

Step 3:

product from step 2



Cinnamaldehyde

Hydrolysis at Room Temperature: While allowing the reaction to continue stirring, slowly add 2M HCl to the reaction at room temperature over the course of 10 minutes. Distill the final product using ether, and then wash with water. Dry the organic layer with sodium sulfate.

Modification: Use magnesium sulfate instead of sodium sulfate (already available in lab)

Green Chemistry and Disposal:

-Potassium hydroxide
\$23.60 / 250g
Harmful if inhaled, swallowed or absorbed through skin. Causes burns. Do not dispose in drains.
-3-Chloropropionaldehyde diethylacetal
\$55.00 / 25mL
Flammable. Irritant. Do not breathe vapor. Dispose with care in a chemical incinerator.
-Potassium carbonate
\$42.30 / 500g
Irritant.
-Bromobenzene
\$43.30 / 50mL
Combustible. Flammable. Irritant. Environmentally dangerous. Cover spills with dry-lime, sand or soda ash. Dispose with care in a chemical incinerator.
-Palladium(II) acetate
\$102.00 / 1g
Irritant. Risk of serious damage to eyes. Contact a licensed professional waste disposal service.
-Hydrochloric acid (2M)
\$19.30 / 1L
Irritant.
-Potassium chloride
\$59.70 / 500g
Mild irritant.