Synthesis of Dibenzylideneacetone by an Aldol Condensation

CHEM HELP *ASAP*

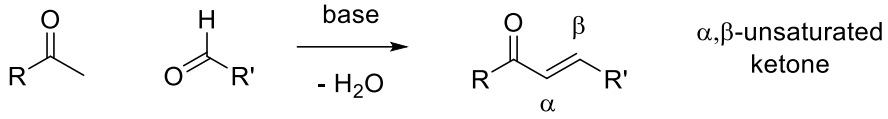
experiment video: https://youtu.be/2YOi5HvFk_A

Purpose

The purpose of this experiment is to synthesize dibenzylideneacetone through an aldol condensation between acetone and benzaldehyde. The crude product will be isolated by simple filtration and is pure enough to be characterized in its crude form.

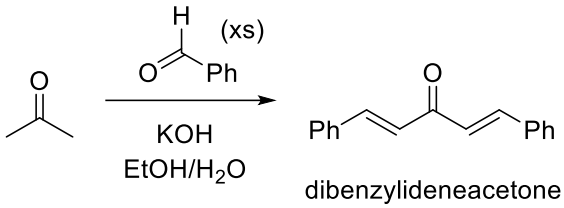
Background

The aldol condensation is a staple reaction for organic chemistry (Scheme 1). The reaction involves the deprotonation of a carbonyl (often a ketone) from its α-carbon to make an enolate. The enolate then attacks another carbonyl (often an aldehyde). Through a series of steps, the final product is an α,β-unsaturated ketone.



Scheme 1. Thermal azide-alkyne 1,3-dipolar cycloaddition

The synthesis of dibenzylideneacetone involves the aldol condensation of acetone and benzaldehyde. The reaction requires two equivalents of benzaldehyde relative to acetone in order to form the final product. The reaction is driven largely by the insolubility of dibenzylideneacetone in the reaction solvent of ethanol and water.



Scheme 2. Today's reaction – aldol condensation of acetone and benzaldehyde

Procedure – dibenzylideneacetone

(adapted from Conard, C. R.; Dolliver, M. A. *Org. Synth.* **1932,** *12,* 22)

In a 125 mL Erlenmeyer flask dissolve approximately 5.0 g of KOH in 30 mL of 1:1 ethanol:water. Prepare a separate solution of acetone (25 mmol) and benzaldehyde (55 mmol) in 30 mL of 1:1 ethanol:water. Add the acetone-benzaldehyde solution in approximately 2 mL portions to the stirring KOH solution over a few minutes. Allow the combined mixture to stir for approximately 1 h. Filter the mixture through a Buchner funnel with a 250 mL side-arm flask. Seat the filter paper with an ethanol-water mixture. Thoroughly rinse the solid with water. Spread the collected solid on a tared watch glass to air dry. Determine the mass of the dry product. Calculate the percent yield. Determine the melting point (melting range – both upper and lower limits) of the product. Take a TLC of the product using 10% ethyl acetate/90% hexane as the mobile phase. Once you finish your TLC, dispose your mobile phase in a waste jug. Interpret the provided NMR spectrum. Record all your observations in your notebook.