In this experiment, an aromatic aldehyde, piperonal, is allowed to react with an excess of acetophenone in the presence of a base. Loss of water from the initial aldol product leads to the formation of a very stable structure, an α,β-unsaturated ketone in conjugation with an aromatic ring.

The starting aldehyde, piperonal, is a naturally occurring compound found in black locust (*Robinia pseudoacacia*), a tree which is majorly used for timber but also for paper pulp, posts and fuel. Black locust wood is also being studied to find the chemical basis for its remarkable decay resistance. Piperonal can also be synthetically made from piperine (*piperylpiperidine*) which is a constituent of pepper.

**Procedure:**
Into a 10 mL Erlenmeyer flask are placed 0.24 g (2.0 mmoles) of acetophenone, 0.30 g (2.0 mmoles) of piperonal (Note 1), 1 mL of 95% ethanol, and 1 mL of 10% sodium hydroxide solution. The mixture is stirred for 30 minutes during which time a solid forms. If an oil forms, it can be induced to crystallize by cooling the reaction mixture and scratching the flask with a glass rod extending into the oil. The reaction mixture is cooled and suction filtered using a Hirsch funnel. The weight of the crude product is recorded in your laboratory notebook and a percent yield calculated for the unpurified product. The crude solid is transferred to a 10 mL Erlenmeyer flask or beaker and recrystallized from a small
amount (2 to 5 mL) of 95% ethanol. To carry out the recrystallization, the crude product is dissolved in a minimum amount of boiling hot ethanol (heating with steam bath) and then allowed to slowly cool to room temperature. After crystals have formed at room temperature, the flask or beaker is cooled in ice and the mixture is suction filtered using a Hirsch funnel to isolate the purified crystalline product. The weight of the recrystallized product is determined and the percent yield is calculated. What percent of the crude material was isolated as recrystallized material? The melting point of the recrystallized product is determined (Note 2). A sample of the purified product is submitted for $^1$H NMR on the second lab day (regardless of whether you ran this reaction on the first or second lab day).

Notes

1. A systematic name for piperonal is 3,4-methylenedioxybenzaldehyde.
2. The reference melting point for 3-(3',4'-methylenedioxyphenyl)-1-phenylpropenone is 122°C.