On treatment with base or acid, aldehydes and ketones with hydrogens on the carbon atom adjacent to the carbonyl group referred to as an $\alpha$-hydrogen can undergo a carbon-carbon bond forming reaction referred to as an aldol condensation.

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 $\alpha,\beta$-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 (*piperyl/piperidine*) 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 tared (weighed) sample vial and recrystallized from a small amount (< 1mL) of 95% ethanol. To carry out the recrystallization, the crude product in the sample vial is dissolved in a minimum amount of boiling hot ethanol and then allowed to slowly cool to room temperature. After crystals have formed at room temperature, the sample vial is cooled in ice and the ethanol is carefully decanted or pipetted out of the vial leaving the crystals behind. The purified crystalline product in the vial is dried by warming the vial over a steam bath to evaporate any residual ethanol. 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) and the remainder of the material in the sample vial is labeled (Note 3) and turned in to the teaching assistant.

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.
3. The label should contain the following information: your name, the name of the compound, the weight of the material in the vial, and the melting point.