# Green microwave catalytic oxidation of benzoin to benzil

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It has been long known that copper (II) acetate is an effective oxidizing agent for the conversion of 2° alcohols to ketones.[1] In this laboratory we will experience this reaction and look to optimize some of the parameters. The overall reaction will be
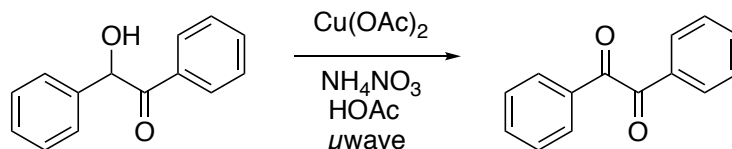
The copper acetate is used in a catalytic amount since it is regenerated by ammonium nitrate. The overall reaction sequence is
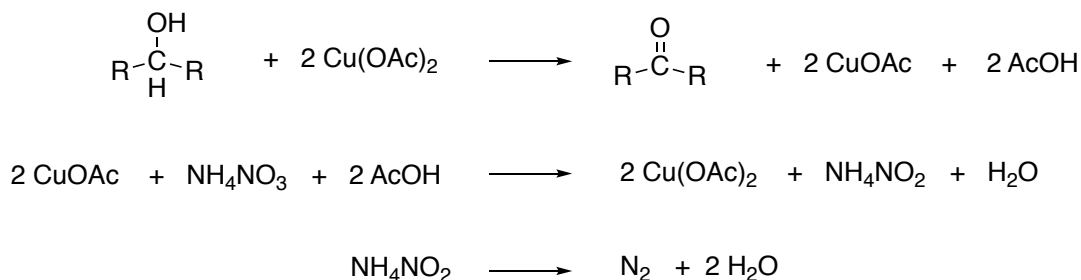
Prelab questions

1. Using the three steps of the overall reaction sequence, write the actual balanced reaction for the conversion of benzoin to benzil.
2. Calculate the atom economy for the conversion of benzoin into benzil.
3. During the procedure a great deal of pressure is developed in the reaction tube. Why?
4. Convert the mass of benzoin from grams to millimoles.
5. Convert the mass of copper (II) acetate from grams to millimoles.
6. Convert the mass of ammonium nitrate from grams to millimoles.
7. Calculate the ratio of benzoin millimoles to copper (II) acetate millimoles.
8. The first step of the reaction sequence shows a 1:2 ratio of benzoin to copper (II) acetate. What's going on?
9. Which of the "12 Principles of Green Chemistry"[2] are addressed in this process?

Procedure

Place a micro stirbar into a microwave tube. Add, in this order, 1.060 g benzoin, 0.500 g ammonium nitrate, 0.010 g copper (II) acetate, and 3.5 ml of 80% aqueous acetic acid. Cap the tube and heat in the microwave reactor for 5-10 minutes at 120-150 °C (see instructor for time/temp settings for your experiment). Note: frequently, the tube must be vented if the pressure exceeds the automatic safety cutoff in the reactor. See your instructor if this happens.

After completion, the tube is cooled at room temperature followed by 10 minutes on ice. The product is collected by vacuum filtration with ice water wash. The crude product is then recrystallized from ethanol.

Set the labelled product aside until next week when you will determine mass, melting point, and IR spectrum. Calculate percentage yield and E factor for the method.

References
1. Weiss, M.; Appel, M. The Catalytic Oxidation of Benzoin to Benzil. *J. Am. Chem. Soc.* **1948**, *70*, 3666-3667.
2. "12 principles of green chemistry" http://www.beyondbenign.org/about-green-chemistry/ (accessed January 10, 2018).