Benzil Synthesis (Experiment XIV-E Part B)

Background Reading

Small Scale Syntheses, pages 26-7 and 62 (Recrystallization)
Zubrick, J.W., The Organic Chem Lab Survival Manual, chapter 13
(Recrystallization. See finding a good solvent and general guidelines.)
Fowler, P., Class Notes: Chapter 12 Part 2 - IR Spectroscopy
(See table of absorptions on page 5.)

Keywords

Oxidation, Dehydrogenation, Recrystallization, and 1,2-Diketone

Compound, Reaction, and Yield Data

- No reaction mechanism is necessary for your lab report.
- For the yield calculations, the molarity of concentrated HNO_3 is 15.8 M, and 1 mole of HNO_3 is needed for each 1 mole of benzil.
- Compare experimental melting range with reference value (94 to 95 °C).
- Interpret peaks present in IR spectrum (see step 7).

Procedure

1. Place 1.0 g of benzoin in a 50-ml beaker.
   Carefully add 3.0 ml of concentrated $HNO_{3(aq)}$ drop-wise and slowly.

   Caution – Concentrated HNO_3 solutions are corrosive and cause acid burns.
   Use gloves and avoid all contact with skin and eyes.

2. Heat mixture in a boiling water bath for 20 minutes.

   Caution – Gaseous oxides of nitrogen (brown in color) are evolved by the reaction.
   These fumes are toxic and corrosive. Perform this step in a hood.

3. Add 30 ml of 10 ^{o}C DI water to the mixture in the beaker. Place the beaker in an ice-water bath, if necessary, to form the crude solid, which is normally yellow in color.

4. Collect the solid by vacuum filtration using a 55-mm Büchner funnel and a vacuum flask.
   Use a small glass beaker to press the aqueous acid out of the crystals.
   Apply vacuum for at least 30 more seconds to fully remove the liquid.
   Dispose of aqueous filtrate in the appropriately-labeled waste jar.

   Caution – Nitric acid can react violently with ethanol.
   Be sure to thoroughly remove of the all aqueous acid from the crystals.

5. Place the solid, along with a stir bar, in a 50-ml Erlenmeyer flask.
   Add just enough 95% ethanol (20 – 30 ml) to dissolve the crystals with stirring.
   Filter or decant to remove insoluble impurities, if any.

6. Place flask in an ice-water bath to crystallize the product.
   If necessary, add 5 ml of DI water to force crystallization.
   Weigh a piece of 55-mm filter paper, and then collect the product by vacuum filtration.
   Dispose of ethanol filtrate in the appropriately labeled waste jar.

7. Dry product in oven at 50 ^{o}C for 10 minutes.
   Then, determine the product mass and melting range.
   Obtain an IR spectrum as well. Label and interpret the IR absorptions (cm^{-1}) at 3060 (med), 2000 – 1700 (weak "bumps"), 1690 (strong), 1660 (med), and 1450 (med).
   Do not misinterpret peaks due to mineral oil if used to dissolve the product.
   Dispose of product in the appropriately labeled waste jar.

Post-Lab Questions

1. Review step 4. What impurity could still be present in step 5?
   What would be the consequence if it was not removed?

2. What oxidation product could result if you mixed ethanol and nitric acid?
   Refer to Oxidation in the Chapter 17 notes.

3. Refer to the Background Reading, and consider what happens to the product in steps 5 and 6.
   What solubility properties make ethanol an ideal solvent for recrystallization?

4. Consider what effect water has on the mixture in steps 3 and 6.
   What would happen in step 5 if you used water as the recrystallization solvent?

5. What peak disappears from the IR spectrum when benzoin is converted to benzil? Why?
   Refer to the two spectra on page 350 in the lab manual. See the Background Reading also.