Oxidation Procedure

- 1. Place 1.0 g of the unknown benzylic alcohol assigned to you in a **100-mL** beaker equipped with a stirbar and add 15 mL of reagent-grade acetone. Stir the solution to dissolve the solid.
- 2. Weigh 85 mg of cuprous bromide (CuBr), 100 mg of 2,2'-bipyridine (BYP), and 100 mg of 2,2,6,6-tetramethylpiperidinyloxyl (TEMPO) into *separate* weigh boats.
- 3. Sequentially add the solid CuBr, then TEMPO, and then BYP to the rapidly stirred solution of the benzylic alcohol. **THIS ORDER IS IMPORTANT!**
- 4. Using a Pasteur pipet, add 10 drops of *N*-methylimidazole (NMI) to the stirred solution. Place a watch glass on top of the beaker and continue stirring the reaction mixture rapidly for 1 hour.
- 5. Pour the solution into your separatory funnel through a glass funnel with a small piece of cotton at the base. Add 20 mL of water to the funnel, swirl the mixture, and then add 20 mL of pentane. Shake the funnel gently, venting as necessary.
- 6. If a precipitate appears, add an additional 10-mL portion of acetone. Shake the funnel gently, venting as necessary, to dissolve the solids. If a precipitate remains, add an additional 10-mL of acetone, and shake the funnel gently, venting as necessary.
- 7. Allow the layers to separate and extract the aqueous layer with an additional 20-mL portion of pentane.
- 8. Combine the organic layers and dry with sodium sulfate. Decant the combined organic extracts to a 150 mL beaker. Store the beaker in your drawer for the following week.
- 9. If there is any liquid left, remove it by air drying it. Add 5 mL of *cold* pentane to the flask, stir the mixture briefly, and isolate the solid product by vacuum filtration. Rinse the solid twice with 5 mL portion of *cold* pentane.
- 10. Weigh the product, determine its melting point, and obtain an IR spectrum.