Bromination of an alkene using pyridinium perbromide to produce a chiral product.

Reading Assignment: CER SYNTH 719.

This experiment demonstrates the bromination of a solid alkene to produce a chiral vicinal dibromide. The product is characterized by MP and chemical tests.

- 1. Preheat the hot plate and aluminum block at a heat setting of ~130-145 °C while you assemble your glassware.
- 2. Put together a microscale reflux set-up (SYNTH 719 ,Fig. 7a). Record the name of the alkene chosen. Use reactant amounts for the microscale procedure. Transfer the reactants to your 5-ml conical vial. Glacial acetic acid has a sharp smell, so dispense it under a hood. Pyridinium perbromide is very corrosive to metal. The alkene should be accurately weighed, since yield is based on its amount. Record the mass to the nearest mg. Add your large magnetic stir bar (to regulate boiling and avoid bumping), and attach the water condenser. Water goes in the bottom of the condenser jacket slowly. Do not let the condenser tubing touch the top of the hotplate. Reflux for 20 min. Start timing after the first signs of condensation, indicating boiling
- 3. Allow to cool, then add water via a pipet down the condenser while stirring rapidly to precipitate the product. Cool in an ice bath to minimize solubility.
- 4. Isolate the product by vacuum filtration using the large, 3.0 cm Hirsch funnel. 3.0 cm Filter paper should be in your common drawer. The product should be white and the filtrate pale yellow. Weigh and determine theoretical yield and % yield.
- 5. Run MP and chemical test on the product: AgNO3 in EtOH, which will produce a turbidity or precipitate in the presence of a halogenated organic compound. The test reagent will stain skin & clothing.
- 6. Tabulate your results in your lab book. Show sample calculations. Comment on the outcome of the experiment.