

## Chemistry 3719L – Lab Session 6

### Synthesis of 4-Bromomethylbenzoic acid by Radical Substitution

#### Pre-lab reading from Zubrick:

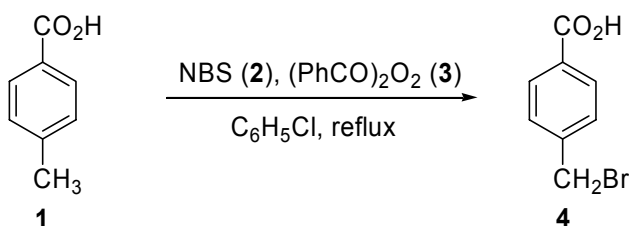
Chapter 23: Pages 201-204 (reflux)  
 Chapter 13: Whole Chapter – review recrystallization  
 Chapter 12: Pages 87-92 – review melting points

#### Aims

This is the first “synthesis” experiment in which a starting material (“reactant”) is converted to a products using “reagents.” You will need to be careful to isolate as much product as possible since it will be used later in the semester as the starting material for another experiment.

#### Reaction

*p*-Toluic acid (4-methylbenzoic acid, **1**) is treated with *N*-bromosuccinimide (NBS, **2**) and a small amount of benzoyl peroxide [(PhCO)<sub>2</sub>O<sub>2</sub>, **3**] to produce 4-bromomethylbenzoic acid (**4**).



#### Procedure

Weigh out 4-methylbenzoic acid (3.00 g, ~15 mmol) and *N*-bromosuccinimide (4.0 g) and add them to a 100 mL round-bottomed flask that contains either a spin bar or a boiling stone. Next add benzoyl peroxide (0.25 g) and then the solvent, chlorobenzene (30 mL) making sure that any solids trapped in the neck of the flask are washed into the flask. (***It is important that there is no benzoyl peroxide left in the ground glass joint as friction between the glass pieces may cause explosion***). Connect the reflux condenser (water in at the bottom, out at the top) and boil the mixture **gently** for 1 hour. Your 1 hour begins when the mixture begins boiling, not when you first turn the heat on. Swirl the mixture every five minutes to ensure that it mixes properly.

After the 1 hour at reflux cool the flask to room temperature and then cool it in an ice bath to allow the product to precipitate. Filter the solids with suction (Zubrick page 109) and wash the solid with portions of hexane (3 x 10 mL) to remove byproducts. Transfer the solid to a small beaker and add DI water (75 mL). Stir the slurry thoroughly to dissolve up succinimide (the byproduct from NBS) and then filter the solid with suction. Wash the solid with water (2 x 15 mL) and hexane (2 x 15 mL) and leave the product to dry under suction for 10 minutes. Obtain a crude weight of the solid material (record in your notebook) and then recrystallize the solid carefully from a minimal amount of ethyl acetate. Again, suction filter the solid, dry under suction, and finally get a weight and melting point for your purified material. Give the product to your TA in a sealed, labeled test tube. It will be stored you will need the material later in the semester.

**Keep good notes to help you write the report. Ask the TA if you don't follow the idea of “per cent yield” since this will be required as part of the report.**