



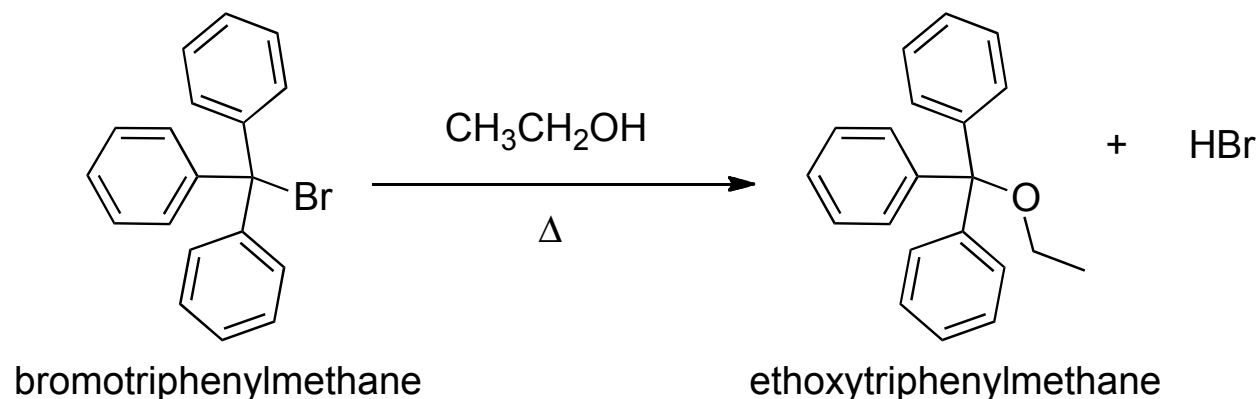
## Experiment 3

### The Williamson ether synthesis

Chemistry 132  
Spring 2013

#### Background

The Williamson ether synthesis yields an ether by the reaction of an alkyl halide with an alkoxide or alcohol. Today you are treating bromotriphenylmethane with ethanol to synthesize ethoxytriphenylmethane.



#### Goals for this week's laboratory include:

1. good technique in setting up a reaction
2. monitoring a reaction to ascertain completion
3. recrystallization of a reaction product

#### Post-lab goals include:

1. interpretation of NMR spectra

### Initial Procedure (work individually)

- Obtain a clean dry test tube.
- Add a boiling chip.
- Add 0.50 g of bromotriphenylmethane to the test tube. (Mass the actual amount of bromotriphenylmethane you add and record this value in your notebook.)

### Reaction (performed in the fume hood)

- Add 5.0 mL of absolute ethanol to the bromotriphenylmethane and boiling chip in the test tube and swirl the reagents to mix them.
- Gently boil the mixture in a hot water bath.
- Heat the mixture until no more HBr evolves or for a minimum of 15 minutes. Test for HBr by holding moistened litmus paper over the mouth of the test tube (use forceps).
- As the reaction heats, replace the ethanol that evaporates, but **do not add a large excess** of ethanol. *This would impede crystallization later!!!!*
- Once the reaction is complete, remove the boiling chip, and let the reaction mixture cool to room temperature (RT).

### Recrystallization

- Crystals may form as the reaction mixture cools to RT. If this happens, then chill the mixture in an ice bath and filter to recover your solid ether product. Rinse your crystals with a minimal amount of chilled water.
- If crystals did not form, try to induce crystallization by gently scratching the test tube. Be careful not to break the test tube – they are fragile! You could also transfer the reaction to a clean scratched Erlenmeyer flask. Also trying chilling the mixture to induce crystallization. If you still do not have crystals, you may need to reduce the solvent volume.

### Analysis

- Mass – calculate a percent yield
- NMR – assign the peaks in your NMR spectra