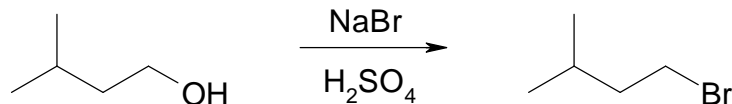


Organic Chemistry Laboratory

#9 - Preparation of 1-Bromo-3-methylbutane



Pre-lab:

Read over the procedure and calculate the theoretical yield of 1-bromo-3-methylbutane.

Procedure:

In a 100 mL round-bottom flask place 11.1 g of sodium bromide and add 10 mL water and 10 mL of the isoamyl alcohol. Mix the contents of the flask thoroughly by swirling; then cool the flask in an ice-water bath. Slowly add 10 mL of concentrated sulfuric acid while keeping the flask in the ice-water. Remove the flask and attach a reflux condenser and place in a ring stand. Heat the flask to a gentle reflux for approximately 45 minutes.

Allow the flask to cool some before removing the reflux condenser and replacing it with a simple distillation setup. Add a couple boiling stones to the flask and place the receiving flask in an ice-water bath. Distill your crude product carefully.

Transfer your crude product to a separatory funnel and add about 10 mL of water. Shake the funnel with frequent venting. Separate the layers and determine which is the organic layer. Wash the organic layer with two 5 mL portions of cold 10 % sodium hydroxide solution. Next wash the organic layer with 10 mL of water and then with 10 mL of saturated sodium chloride solution. Transfer the organic layer to a clean, dry Erlenmeyer flask and dry it with anhydrous sodium sulfate. After about 10 or 15 minutes the product should be clear, if not add some more sodium sulfate.

Check the purity of your product by IR and refractive index. If your product is not pure then transfer your dry product to a clean, dry 25 mL flask and fit the flask for a simple distillation. Place an ice-water bath around the receiving flask. Cautiously distill the product. An accurate boiling point may be difficult to obtain.

Place your product in a clean, dry test tube fitted with a cork and sealed with parafilm. Label your product with your name, product name, and weight in grams. Turn in your product to your instructor before you leave.