Preparation of 1-Bromobutane

The mechanism is $S_N2$. The overall reaction is:

$$\text{H}_2\text{SO}_4 + \text{NaBr} + \text{CH}_3\text{CH}_2\text{CH}_2\text{OH} \rightarrow \text{6 CH}_3\text{CH}_2\text{CH}_2\text{Br} + \text{H}_2\text{O} + \text{NaHSO}_4$$

Boiling Point 118°C  Boiling Point 92°C

At your hood workstation, place a hotplate/stirrer atop a ringstand.

Take a 100-mL round-bottomed flask, and clamp the flask to the ringstand, leaving about 1 inch of room between the bottom of the flask and a plastic bowl, which sits atop the hotplate/stirrer.

Add 17 gm. Of NaBr, 17 mL H$_2$O, and 10 mL 1-butanol to the flask and begin stirring the solution with the magnetic stirrer.

Add a slurry of ice and water to the bowl beneath the flask.

**S L O W L Y AND CAREFULLY** add 14mL conc. H$_2$SO$_4$ to the flask.

Remove the ice bath and replace it with a heating mantle.

Add two or three boiling chips to the flask. Equip the flask with a reflux condenser and begin circulating water through it, water going in the bottom and out the top of the condenser jacket (see Figure on next page).

Heat the flask gently (using a heating mantle setting of about 50% of capacity).

Continue gentle heating until the mixture begins to reflux.

**Once this is observed, continue "refluxing" the mixture for 60 minutes, controlling the level of the vapor in the condenser so that it reaches no more than half-way up the condenser**

At the end of the 60 minute reflux period, discontinue heating ("drop" the heating mantle from beneath the condenser by lowering the metal ring( or lab jack ) holding the mantle to the base of the flask).
Once the flask has air-cooled for a few minutes (3 – 5), disconnect the flask and pour the contents into a 125mL separatory funnel.

Separate the lower aqueous layer from the organic layer and add it to a 100mL beaker containing 50mL H₂O. If the contents of the beaker is one miscible, single layer, add it to 700mL H₂O in a 1 L Beaker, WITH STIRRING, otherwise, consult your instructor.

Extract the organic layer with 14 mL 9M H₂SO₄.

Separate the lower aqueous layer from the organic layer and add it to the contents of your 1 L Beaker, WITH STIRRING.

Extract the organic layer with 14 mL H₂O.
Separate the *UPPER* aqueous layer from the organic layer and add it to the contents of your 1 L Beaker, **WITH STIRRING**.

Extract the organic layer with 14mL saturated sodium bicarbonate.

**Drain the lower ORGANIC layer into a DRY 50 mL Erlenmeyer flask, and add 1gm. CaCl₂.** Swirl the flask occasionally for a period of 5 minutes.

Allow the drying agent to settle and **DECANT** the liquid into a TARED sample bottle, **appropriately labeled**. Save it in your locker for the alkyl halide tests.

**Notes:**

Steps 4-8: The reaction, with mechanism is:

\[ n-\text{C}_n\text{H}_2\text{O} + \text{H}^+ \rightarrow n-\text{C}_n\text{H}_2\text{OH} \]

Steps 7-13: Initially, the reaction is exothermic which makes the reaction occur, but quickly loses the energy, so the energy must be supplied by the heating mantle in order to complete the reaction as much as possible.

Refluxing is a means of trapping the vapors of the reactants and cooling them enough to return to the reaction flask. There, they have another opportunity to react to form 1-bromobutane.

Steps 17-20: To separate the 1-bromobutane, a series of extractions are performed. To determine which layer is the 1-bromobutane layer, you take a test tube and add 5 mL of water to it, then remove a few drops of the bottom layer from the separatory funnel and see if they fall through the water to the bottom. If they do, the bottom layer is the 1-bromobutane layer or organic layer, if not, the upper layer is the organic layer. To confirm your decision, remove a few drops of the top layer in the separatory funnel with a pasteur pipette and place them in a dry test tube and add 2 to 3 mL of water, dropwise, to see if it falls to the bottom. The larger layer should be the water layer.

Step 23: Washing the organic layer with 9M H₂SO₄ removes any unreacted 1-butanol.
Step 25: Washing with water simply removes any unreacted 9M H$_2$SO$_4$ molecules.

Step 26: Washing with saturated NAHCO$_3$ removes any remaining 9M H$_2$SO$_4$ molecules.

Step 28: The water is removed by CaCl$_2$, as described in the propanoic acid experiment.
Title: Synthesis of 1-Bromobutane

Name: 

Date: 

Equation:

Table of Physical Constants

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Results: Calculate the % yield for 1-Bromobutane

Boiling range for your product __________ to __________ Deg.C.