Preparation of 2-Chloro-2-methylbutane

Reading Assignment:

1) Hand out sheet pages 1-2

Include in your report:

Reaction mechanism reaction, actual yield, theoretical yield, percent yield, and boiling point (experimental and literature).

Important Data:

1. Actual yield of 2-Chloro-2-methylbutane

2. Theoretical yield of 2-Chloro-2-methylbutane

3. Percent yield of 2-Chloro-2-methylbutane

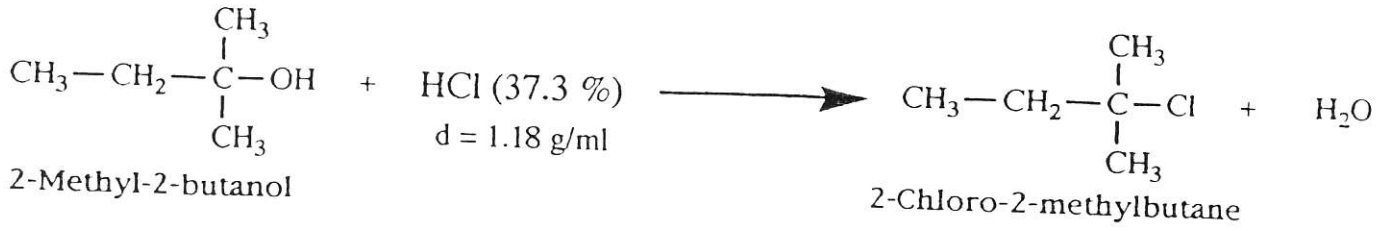
4. Boiling point of 2-Chloro-2-methylbutane

Questions:

Answer questions #1-5 Handout sheet

Preparation of 2-Chloro-2-methylbutane

2-Chloro-2-methylbutane is prepared by reacting 2-Methyl-2-butanol (*t*-amyl alcohol) with hydrochloric acid. The overall equation is given below:



2-Methyl-2-butanol

2-Chloro-2-methylbutane

A. Reaction Procedure

Charge 22 mL of 2-Methyl-2-butanol (*t*-amyl alcohol) to a 125 mL separatory funnel. Add 50 mL of concentrated hydrochloric acid. **Caution, do not stopper the funnel!!!**

Mix the reacts together by gently swirling the mixture in the separatory funnel for several minutes. After several minutes of swirling, stopper the separatory funnel and invert it. Without shaking the separatory funnel, carefully open the stopcock and vent out the pressure. After venting the pressure, close the stopcock, shake the funnel several times, and vent the pressure through the stopcock a second time. Continue to shake the funnel for 3 to 5 minutes, venting the pressure periodically. After 5 minutes of shaking and venting, allow the reaction mixture to stand in the separatory funnel until two layers completely separate. Separate and save the crude organic product layer.

B. Wash Procedure (Extraction)

The wash procedure should be done as quickly as possible since the 2-Chloro-2-methylbutane is unstable in water and sodium bicarbonate solution.

First, make sure the crude organic layer is in the separatory funnel. Do a water wash on the organic layer by adding 25-mL water of to the crude organic layer in the separatory funnel water. Be sure to swirl and shake the two layers together so that a good water extraction of impurities is achieved.

After you have washed the organic layer, allow the layers to separate and discard the aqueous layer. Make sure you know which layer is the organic product layer.

Next, base wash the organic layer by adding 25-mL of 5% aqueous sodium bicarbonate to the organic layer in the separatory funnel water. Be sure to swirl and shake the two layers together so that a good base extraction of impurities is achieved. Gently swirl the funnel (unstoppered) until the contents are thoroughly mixed. Stopper and invent the separatory funnel. Carefully vent the excess pressure through the stopcock. Gently shake the separatory funnel, with frequent release of pressure.

Preparation of 2-Chloro-2-methylbutane (continued)

Repeat this procedure of stoppering the funnel, shaking, and venting pressure for about one minute. Allow the layers to separate, drain off and discard the lower aqueous bicarbonate layer.

Finally, water wash the organic layer by adding 25 mL of water to the organic layer in the separatory funnel. Shake and swirl and shake the two layers together so that a good water extraction of impurities is achieved. After you have washed the organic layer, allow the layers to separate and discard the aqueous layer.

Transfer the washed organic layer to a small dry 50 mL Erlenmeyer flask. Dry the crude 2-Chloro-2-methylbutane by adding 2 grams of anhydrous calcium chloride. Swirl the product with the drying agent until the liquid becomes clear. Additional anhydrous calcium chloride may be required if the liquid does not clarify. Only add 0.500 gram increments of anhydrous calcium chloride.

C. Distillation Procedure

Decant (or gravity filter) the clear dried liquid into a 100 mL **dry** distilling flask. Add a magnetic stirring bar (or boiling stone) and distill the crude 2-Chloro-2-methylbutane in a **dry** simple distillation apparatus using a water bath. Make sure the thermometer placement is correct. Distil the crude dried product, and collect the pure 2-Chloro-2-methylbutane in a receiver cooled in an ice bath. The final product boils between 79 and 84 °C. Obtain the actual yield of the purified product. Submit the sample to the instructor in a properly labeled vial.

D. Questions

1. Why is the crude product washed first with water? Hint: Look up the solubility of 2-Methyl-2-butanol and 2-Chloro-2-methylbutane in water.

2. Why is aqueous sodium bicarbonate used to wash the crude 2-Chloro-2-methylbutane. Why would it be undesirable to wash the crude product with aqueous sodium hydroxide?

3. The crude product is washed a second time with water. Why?

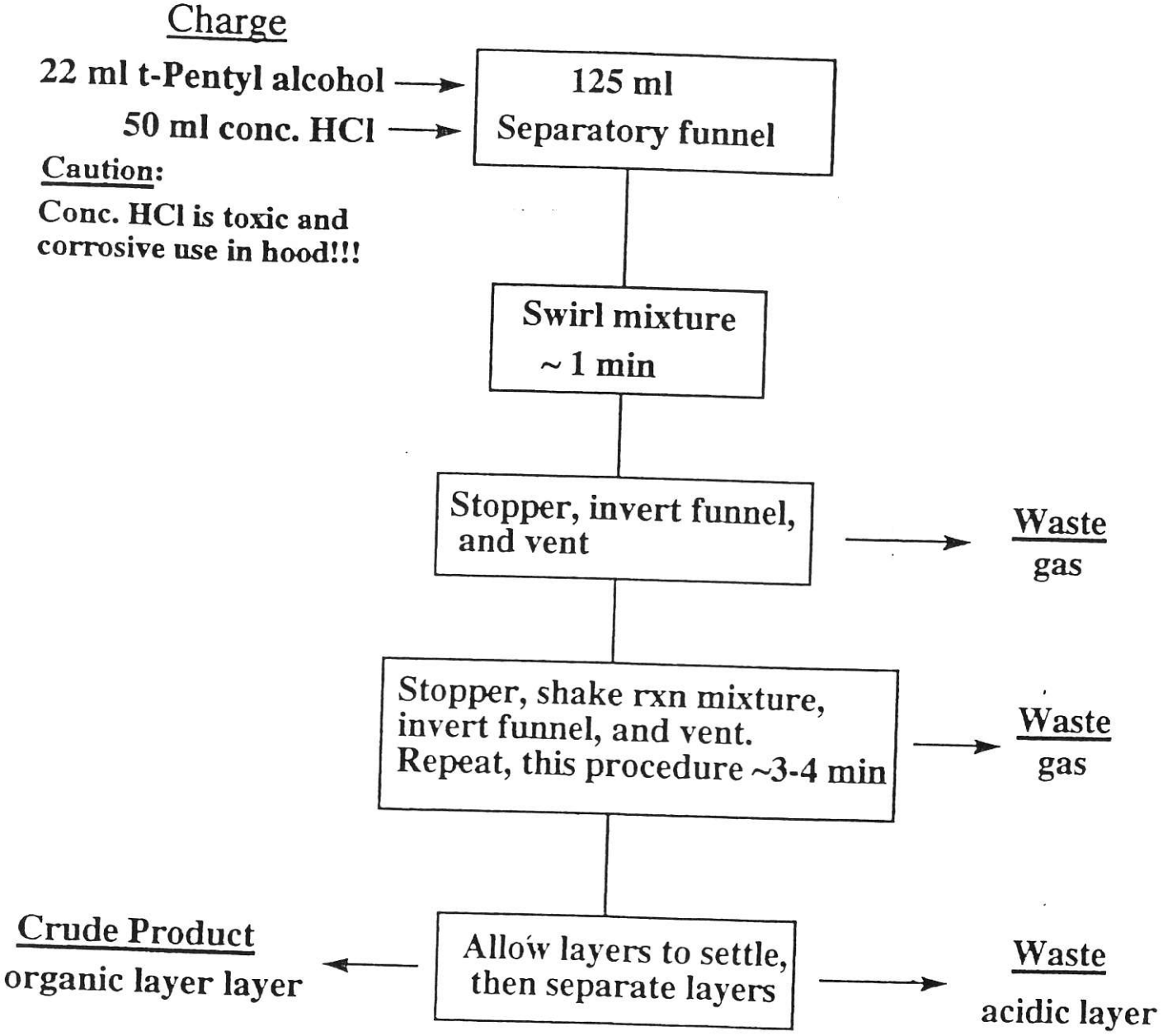
4. Why must the crude 2-Chloro-2-methylbutane be dried carefully with anhydrous calcium chloride before the final distillation?

5. a) A small amount of 2-methyl-2-butene may be produced in the reaction as a side-product. Write out the mechanism for 2-methyl-2-butene formation.

   b) How is the 2-methyl-2-butene impurity removed from the product?

Preparation of t-Pentyl Chloride

Reaction Flow Diagram and Isolation of Crude Product:



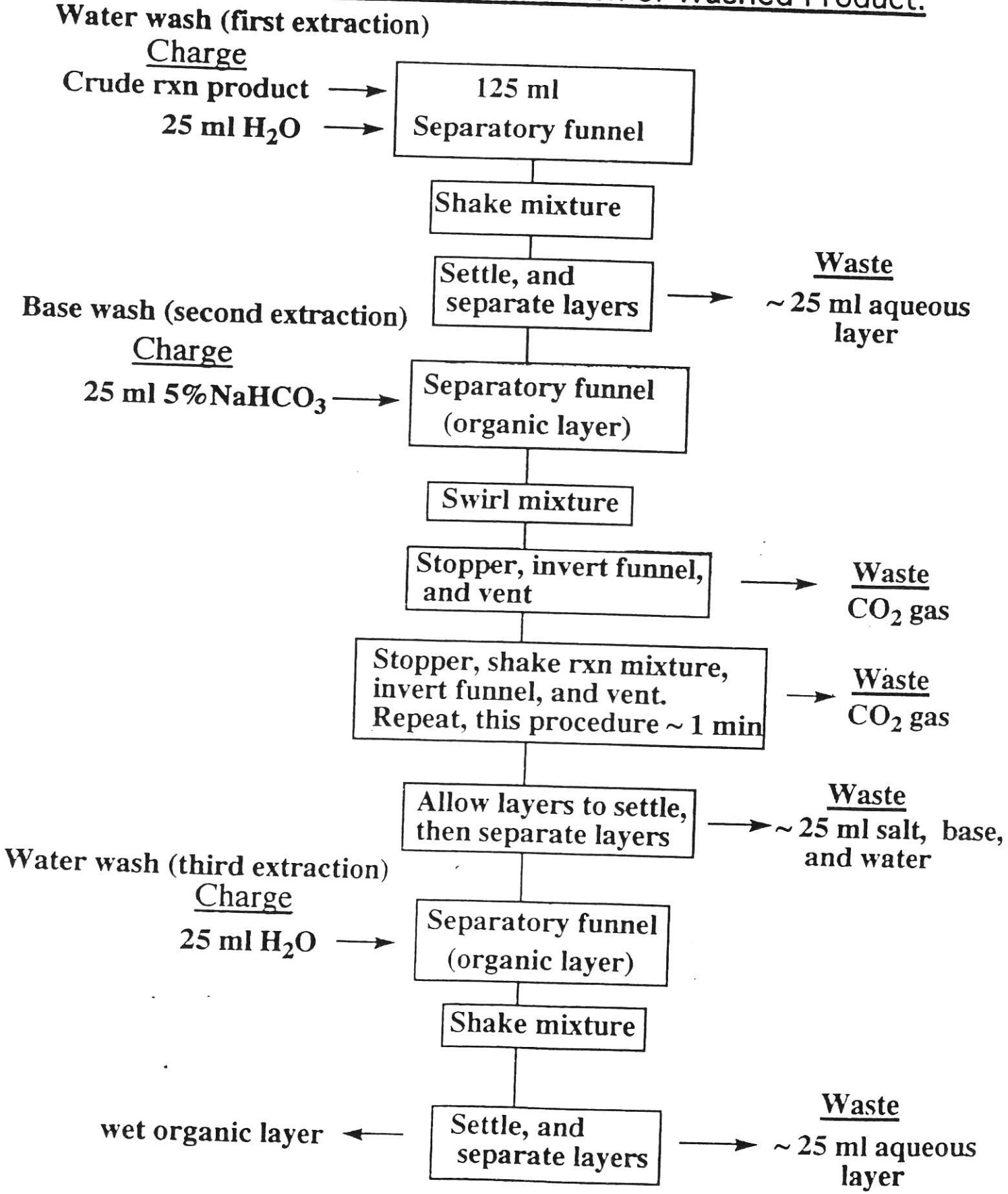
How do you know which layer to save? Top or bottom layer?

Hint: { density $H_2O \sim 1$ g/ml
density $C_5H_{11}Cl = 0.865$ g/ml

Solubility
"Likes dissolve likes"

Preparation of t-Pentyl Chloride

Extraction Flow Diagram and Isolation of Washed Product:



Preparation of t-Pentyl Chloride
Drying & Distillation Flow Diagram