

## SIMPLE AND FRACTIONAL DISTILLATION

I. PURPOSE: To learn how to distill a miscible liquid mixture smoothly and efficiently and to understand the limits of separation by distillation.

II. ACTIVITIES:

Read : Landgrebe pp 142-157

We will work in pairs for this experiment. You may select your own partner. Each pair will perform each of the following types of distillation: (a) Simple distillation, (b) Fractional distillation - open column, (c) Fractional distillation - ceramic saddles. This experiment will take two lab periods to complete. You should complete one distillation the first period and two the second period.

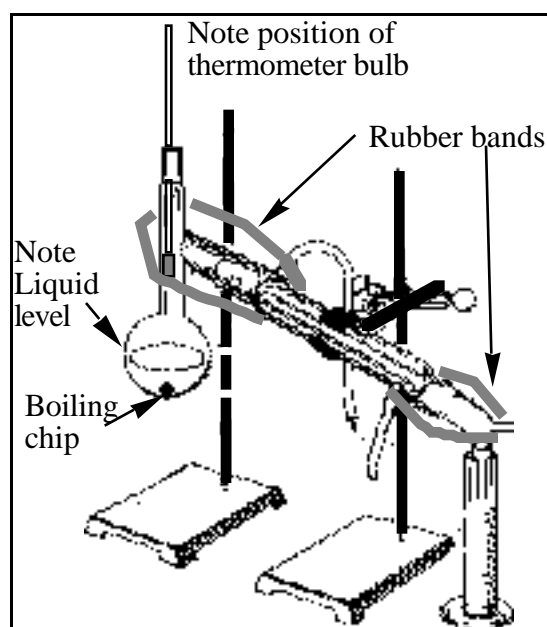
It will be necessary to regulate the boiling rate and read the temperature and measure the volume distilled. Record, for your initial temperature, the temperature after the first five drops distill and after each two mL of distillate is collected. Record the data on the attached data sheet.

As soon as the distillation is complete, transfer your data to the table on the blackboard at the front of the lab. Before you leave the lab, be sure to record ALL the data from the other distillation experiments. You will need to average all the data for each type of distillation.

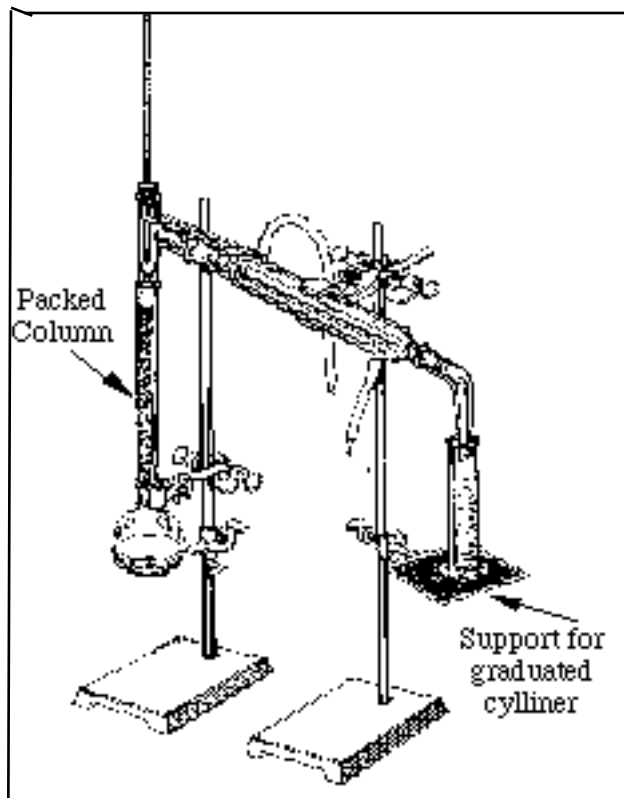
During the second lab period, we will learn about the graphical presentation of data and plotting your data using the Macintosh computer. You will plot, on one graph, the average boiling temperature (y axis) vs the milliliters distilled (x axis) for each type of distillation. Use a different color or type of line to differentiate between the plots. All axes and curves must be clearly labeled.

IV. APPARATUS:

Simple distillation: The set-up is shown on page 65 except that the water cooled condenser will be allowed to drip directly into a graduate cylinder.



- (a) Choose the distilling flask so that it is initially 30 to 60% full. We will distill 40 ml of a 50:50 mixture of cyclohexane and toluene (20 mL of cyclohexane and 20 mL of toluene) so use your 100 ml flask.
- (b) Do not lubricate the joints. Ground glass joints should be lubricated only for reduced pressure distillation. At atmospheric pressure solvent enters the joints and dissolves out the lubricant leaving a solid residue which may freeze the joints.
- (c) It is customary to distill from left to right.
- (d) Support the thermowell heater on a ring clamp leaving enough space under the ring to allow lowering of the heater. Use 2 condenser clamps, one to support the distillation flask at its neck and one on the water cooled condenser. Collect the product in a 10 mL graduated cylinder attached with a condenser clamp on a second ring stand. Adjust the height of the graduated cylinder so that it is just under the condenser outlet.
- (e) Place a Boileaser chip in the flask to prevent bumping.
- (f) Note the thermometer position in the figure on page 65. The top of the thermometer bulb should be slightly below the bottom of the side arm of the distilling head to accurately measure the distillation temperature.
- (g) Cooling water should enter at the bottom inlet of the horizontal condenser and exit at the top inlet.

Fractional distillation:

- (a) Use the wide-bore column as the vertical column ( in the fractional distillation). Use the narrow-bore column for the horizontal condenser (all distillations).
- (b) The vertical column of the fractional distillation set-ups should be packed loosely with glass wool or ceramic saddles and wrapped with aluminum foil. If the column is packed too tightly, it will flood (fill up with liquid) during the distillation and you will not separate the liquids. The vertical column must NOT be water cooled. it is appropriately call an air cooled fractionation column. Columns specifically made for this purpose are shown on page 73.
- (c) Get your lab instructor to approve your set-up before you begin your distillation.

## SAFETY PRECAUTIONS:

When you are ready to start the distillation, check the following items. Then ask your lab instructor or assistant to approve your set-up. Failure to do so could result in an accident.

1. A boiler should be in the distillation pot.
2. All joints should fit together without being under tension.
3. The distillation flask should be not more than 3/4 full.
4. No tight plugs of glass wool should occur in the fractionation column.
5. The top of the thermometer bulb should not touch the glassware and should be even with the bottom of the distillation head side arm. The thermometer should fit tightly in the rubber holder.
6. The collecting vessel (10 ml grad. cylinder) should be close to the exit from the condensing column.
7. Make sure that the vapor from the distillation flask can escape to the atmosphere at the end of the apparatus (No closed System).
8. The heating mantle must be able to be lowered quickly to remove the heat source without disturbing the integrity of the distillation apparatus. If, at any time during your distillation, you have doubts about the safety of your operation you should remove the heat source. Not much can happen without it.

## V. PROCEDURE

1. When you are ready to distill, turn the thermowell control to near the highest setting. Reduce the power when the liquid begins to boil so that it distills at a rate of approximately 1 drop per second. It will be necessary to increase the power slightly during the distillation as the lower boiling component is removed and the distillation temperature increases.
2. Record the temperature when the first 5 drops of liquid fall from the condenser into the graduated cylinder. Continue to record the temperature after each successive 2 ml of distillate is collected. Check often to see that the column does not flood. If it starts to fill with liquid, reduce the heating rate.
3. Collect the following 3 fractions during the distillation in labeled, stoppered glass vials provided by the assistant. Save all three fractions for analysis in the next experiment. Be sure they are securely stoppered or they will evaporate during the week.

- Fraction 1 - First 2 ml of distillate
- Fraction 2 - Next 10 ml of distillate
- Fraction 3 - Residue in the pot

Never distill to dryness. Leave about 1 ml of residue. Failure to do so could result in an explosion or a cracked distillation flask.

4. Discard any distillate which is not to be save in the waste bottle provided for this purpose. Never pour organic liquids which are flammable and immiscible with water down the drain.

### III. LAB REPORT:

\*\* Even though you worked in pairs, the lab report should be an individual effort. \*\*

1. Title Page (your name and title of experiment)
2. Experimental Description. Explain the experimental procedure(s) in your own words. Do Not just parrot the lab handout. Indicate your partner on this page
3. Observations. Record any changes or problems which occur during the distillation.
4. Attach the data sheet to your report. Report your individual data as well as the full class data
5. Attach your graphs of boiling point versus volume of distillate for all four types of distillation.
6. Attach your answers to the questions at the end of this handout.

### VI. QUESTIONS:

1. Arrange the fractionation types in their order of increasing efficiency. Using your plot, state specifically how the plot leads you to the above conclusion. This should require about a half a page of discussion.
2. Indicate which types of distillation technique you performed. What is the boiling point of pure cyclohexane and pure toluene? Look them up in the "Handbook of Chemistry and Physics". At what temperature did your distillation begin? Considering this, is it likely that your initial distillate contained only cyclohexane? Explain. What was the temperature at which you stopped our distillations? Considering this temperature, is it likely that this fraction contains only toluene? Explain.

3. The fractionation column makes possible a more complete separation of these liquids than could be obtained in a simple distillation. Describe what happens to the distillate on its way up the fractionation column that improves the separation.
4. Refer to the boiling point diagram for butanol/acetone shown below. Compound A = acetone; B=butanol
  - a. At what temperature would a solution containing 0.5 mole fraction butanol and 0.5 mole fraction acetone begin to boil? How does this temperature compare with the boiling points of the pure compounds?
  - b. What would be the composition of the first droplet of vapor which recondenses? How does this compare to the composition of the liquid being distilled (0.5 mole fraction in each component)? At what temperature will the droplet boil? Will the vapor around the droplet be hot enough to cause the droplet to boil?
  - c. How many times would you need to condense and re-evaporate the first vapor to obtain pure acetone?

