Experiment 4: FRACTIONAL AND SIMPLE DISTILLATION

Experimental Notes and Warnings
1 Exercise care when you put in your thermometer into the thermometer adapter
   i) Hold the thermometer adapter as well as the thermometer using paper towels. Hold the adapter
      with your fingers. **DO NOT PRESS INTO THE PALM OF YOUR HAND.**
      ii) Slide the rubber stopper containing the thermometer with an even pressure on top of the glass
      adapter.

If any rubber tubing, corks or stoppers which have become hardened after excessive use, simply cut the rubber or cork from the glass.

2. The water flow through the condenser **should not be with high force!**
3. Don’t let all the solvents in the distillation flask (RB-flask) go completely dry.
4. The heating mantle needs to be plugged into a transformer.

**Part A. Simple Distillation**

**PROCEDURE**
Assemble the simple distillation apparatus shown below. Use a 100 ml graduated cylinder as you receiving vial. Make sure that the mouth of the grad-cylinder is placed into the mouth of the adaptor/ vacuum-adaptor. Add 25mL of cyclohexane and 25mL of toluene to the round bottom flask, and a couple of boiling chips/boileezers. The thermometer bulb needs to be placed right below the side arm of the distill head.  **Make sure that ring stand is ~ 10cm above the bench-surface of you. This is done to ensure that you can lower the heating in case of over boiling of the liquid mixture.** The distillation apparatus should not be completely closed (ie. There should be some space at the mouth of the vacuum adaptor to vent excess pressure).

![Simple Distillation apparatus](image)

**Simple Distillation apparatus**
Connect the mantle to the transformer (After making sure all connections are tight, heat the flask until boiling starts, and the transformer setting initially should be ~3.5. Record the temperature
every 2 mL as the distillation proceeds till you collect 44-45 mL of distillate. Once ~24-25mL of distillate is collected, empty the graduated cylinder to separate the two liquids and increase the transformer setting to 4.5-5. Continue collecting liquid as a running total. Boileezers/bubbling-chips are used to prevent uneven boiling and prevents "bumping" These are white in color and inert. They contain small pores which has air trapped in. During boiling the air trapped inside the pores escape as small bubbles.

**Part B. Fractional Distillation**

**PROCEDURE**

Assemble the apparatus shown above. Use a 100 ml graduated cylinder as you receiving vial. Make sure that the mouth of the grad-cylinder is placed into the mouth of the adaptor/vacuum-adaptor. Add 25mL of cyclohexane and 25mL of toluene to the round bottom flask, and a couple of boiling chips/boileezers. The thermometer bulb needs to be placed right below the side arm of the distill head. Make sure that ring stand is ~10cm above the bench-surface of you. This is done to ensure that you can lower the heating in case of over boiling of the liquid mixture. The distillation apparatus should not be completely closed (ie. There should be some space at the mouth of the vacuum adaptor to vent excess pressure). Connect the mantle to the transformer (After making sure all connections are tight, heat the flask until boiling starts, and the transformer setting initially should be ~4.5. Record the temperature every 2 mL as the
distillation proceeds till you collect 44-45 mL of distillate. Once ~24-25ml of distillate is collected; increase the transformer setting to 6.

Please also note:
1-The column should be vertical and care should be taken to ensure that the bulb of the thermometer does not touch the side of the distilling head.
2-Gradually turn up the heat to the electric flask heater until the mixture of cyclohexane and toluene just being to boil. As soon as the boiling starts, turn down the power. Heat slowly at first. A ring of condensate will rise slowly through the column; if you cannot at first see this ring, locate it by cautiously touching the column with the fingers. The rise should be very gradual, in order that the column can acquire a uniform temperature gradient. Don't apply more heat until you are sure that the ring of condensate has stopped rising.

Results (both simple and Fractional Distillation)

Record data in a chart (you will need to copy this to your lab notebook). If you are doing simple distillation, then obtain data from a group that did fractional distillation and record values in chart. If you did fractional distillation then, obtain data from a group that did simple distillation and record values in chart.

| Volume (ml) | 2 | 4 | 6 | 8 | 10 | 12 | 14 | 16 | 18 | 20 | 22 | 24 | 26 | 28 | 30 | 32 | 34 | 36 | 38 | 40 | 42 | 44 |
|-------------|---|---|---|---|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|
| Temp (°C)   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Simple      |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Temp (°C)   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Fractional  |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |

Plot both sets of data in one Excel graph. In your note book, comment on the following: On the basis of the graphs/results, which procedure was more efficient in separating the mixture into separate components? Turn in your graph with the lab report.

In your discussion/conclusion; explain in detail what you see in both the simple and fractional distillation curves (explain the results in detail to get any credit!).

POSTLAB QUESTIONS

1. a) Define the term boiling point.
   b) What effect would a reduction in the atmospheric pressure have on the boiling point of a liquid?

2. Why would it be dangerous to heat an organic compound in a distilling apparatus that was closed tightly at every joint and having no vent or opening to the atmosphere or to a vacuum pump?