Experiment V-E (continued): Fractional Distillation

Background Reading

Zanger M., and McKee J.R. Small Scale Syntheses. pp 21-24 (fractional distillation).
Zubrick, J. W. The Organic Chem Lab Survival Manual. fractional distillation in Ch 20.

Key Words

azeotrope (low and high boiling), mole fraction, vaporization, condensation, theoretical plate, boiling curve (vapor-liquid equilibrium), Raoult's Law, and Dalton's Law.

Experimental Data

- Plot temperature (± 0.5°C) vs. total quantity of liquid (0.5 ml increments).
  Depict all three cuts sequentially on one graph. Label the plateaus and steep rises.
  Use the graph to determine true cut sizes, and compare with what you collected.
- Record temperature range for all three cuts, and compare with reference values
  for the substances' boiling points.
- Record refractive indices for the 2nd and 3rd cuts to four decimal places,
  and use the refractive indices spreadsheet to determine compositions.
- Include refractive index for each of the three components in the Substances section.
- Calculate recovery % for each cut based upon liquid volumes (initial divided by final),
  and compare with simple distillation.
- Compare purities and refractive indices for simple vs. fractional distillation labs.

Procedure

Bring simple distillation procedure with you to lab. This experiment is nearly identical nearly to simple distillation, but is modified with a **packed column** to improve separation. The surface area of the packing provides a much larger surface area where liquid and vapor can reach equilibrium. Each effective liquid-gas equilibrium functions as a separate mini-distillation, and is called a theoretical plate. So, the result is a multi-stage distillation.

Assemble fractional distillation apparatus with a packed column between the round-bottom flask and distillation head. Create the packed column by filling a condenser with glass beads. Note location of prongs inside the bore of the condenser. These prongs will hold the beads in place. Be sure to clamp column along with flask and condenser. Also, apply thick insulation with no gaps, because **reduction of heat loss is much more critical** in this experiment.

When experiment is complete, **collect the glass beads** in a beaker,
so that they can be cleaned and reused.

Post-Lab Q's

1. According to the lab manual or the survival manual, when does the difference in boiling points make it necessary to use fractional distillation rather than simple distillation?

2. What would happen to the mole fractions of the separated fractions if the distillation rate was increased? Compare the values of the refractive indices for the three components. How would the refractive indices for each of the three fractions change as a result?

3. What does the packing provide inside the fractionating column?
   How does the packing improve the separation?

4. What would happen without insulation on the outside of the fractionating column? How could your final fraction be affected?

5. Why is the volume of the final distillate fraction obtained for fractional distillation usually less than that obtained for simple distillation?