Experiment V-E: Simple Distillation

Background Reading

Zanger M., and McKee J.R. Small Scale Syntheses. pg 15 (1^{st} ¶ of section B, and Fig 2.1), pp 17-18 (refractive index), and pp 19-21 (simple dist.).

Zubrick, J. W. The Organic Chem Lab Survival Manual. simple distillation, example, & mistake in Ch 20.

Key Words

distillation, mole fraction (or %), vaporization, condensation, normal boiling point, distillate, refractive index, 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 2^{nd} and 3^{rd} cuts. Record indices to four decimal places. Then, use the refractive indices spreadsheet to determine the 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).
- Include diagrams with labels and titles for the glassware and the refractometer.

Substances

8 mL methylene chloride, 8 mL cyclohexane, 8 mL p-xylene, boiling stones

Apparatus

Items in kit
50-mL round-bottom flask
distillation head (three-way adapter)
thermometer adapter (glass with rubber)
condenser
vacuum adapter (leave side nozzle open)

Items not in kit
heating mantle, thermometer, two water hoses, two stands with clamps
three 10-ml graduated cylinders, two small test tubes

Procedure

1. Obtain three 10-ml graduated cylinders.
   Label each for use with one of the three substances.

2. Obtain 8 ml each of methylene chloride (BP 40 ^{o}C), cyclohexane (BP 80 ^{o}C), and p-xylene (BP 138 ^{o}C). Mix together in a 50-ml round bottom flask. Note that flask is approximately half-full. This is ideal as a heated flask should never be more than two-thirds full. Add two or three boiling stones.

3. Assemble simple distillation apparatus. Note that water flows into the condenser near the receiver and out near the distillation head. Use a rubber adapter, and its glass joint, with the thermometer, and carefully place the thermometer tip just below the neck of the flask. Ensure that all glass joints are securely fastened so that vapors will not leak out. Then, wrap the distillation head with aluminum foil as insulation to reduce heat loss. Apparatus must be clamped at the neck of the flask as well as at the condenser.

4. Turn on controller and set controller's dial to approximately 4 in order to begin distillation. Adjust setting as needed, but use small adjustments so as not to overcompensate. In general, the dial will need to be adjusted upwards over the course of the experiment. The heat rate should be adjusted so that the distillation rate is no more than one drop every two seconds. A slower distillation rate will provide a better separation, and result in fractions with higher purity.

   Caution – The apparatus is now hot. Avoid direct contact with glassware and clamps.

5. Place the methylene chloride cylinder at the condenser's outlet and begin collecting the first fraction. Collection T will stay nearly constant at 40 ^{o}C or lower as methylene chloride distills.

6. Record T and total ml of distillate in 0.5 ml increments for use in graphing the results.

7. When the methylene chloride fraction has completely distilled, the T will rise sharply. When this happens, replace the graduate cylinder with the cylinder for cyclohexane. This will happen at or before 8 ml have been collected.

8. During the cyclohexane fraction, the T will stay nearly constant at 80 ^{o}C or lower.
   When the total distillate (sum of volume in both cylinders) is between 10 and 12 ml, obtain approximately 0.5 ml in a small test tube.
   Determine refractive index of the sample on the refractometer.
   Use refractive indices spreadsheet to find mole fraction of cyclohexane.

9. When the cyclohexane fraction has completely distilled, the T will rise sharply. When this happens, replace the graduate cylinder with the cylinder for p-xylene. This will happen before 16 ml have been collected.

10. During the p-xylene fraction, the T will stay nearly constant at 138 ^{o}C or lower. When the total distillate (combined volume of all cylinders) is between 16 and 20 ml, obtain approximately 0.5 ml in a small test tube. Determine refractive index of the sample on the refractometer. Use refractive indices spreadsheet to find mole fraction of p-xylene.

11. Discontinue heating when total volume collected reaches 20 ml or earlier if there is very little liquid left in the distillation flask.

    Caution - Never distill to dryness as the temperature can increase extremely rapidly. Excessive heating can char or break glassware, release dangerous fumes, or create an explosive mixture of vapors, including organic peroxides.

12. Record volume collected for each fraction of distilled liquid. Then, place all used chemicals in the appropriately-labeled waste container(s).

Post-Lab Q's

1. What is the suggested collection rate for the distillate? What might happen if the distillation is carried out at a faster rate?

2. Does the pot temperature increase linearly throughout the distillation? Describe the temperature change rates that are normally observed.

3. Explain how the solubility and volatility of a solution determines whether or not simple distillation is possible.

4. Explain whether distillation be a practical method for removing small amounts of water from an organic liquid. Review at least one of the following sources: page 29 (chapter 2, section II-E) of Zanger and McKee, chapter 10 of Zubrick (Survival Manual), or steps 10 and 11 of the Extraction Lab's procedure.

5. Describe the potential consequences of taking a distillation to dryness. Explain how to prevent such an issue from happening.