Rowan College at Burlington County

CHE 241

Lab 1: Simple Distillation

Objectives:

1. Set up a distillation apparatus
2. Separate a two-component mixture by simple distillation
3. Determine whether separation of the two liquids occurs
4. Determine percent recovery of the two liquids
5. Determine efficiency of the separation

DIRECTIONS-SIMPLE DISTILLATION

In this experiment, simple distillations of three liquid mixtures, with three different boiling point separations, will be done. The percent recoveries and the efficiency of each distillation will be determined. General - all distillations: Before lab, determine the "rule of thumb" fractions that should be collected and record them in the Data Table. If these overlap, no pure fractions can be collected and the entire volume is collected as Fraction I. The position of the mercury bulb of the thermometer is very important; the entire bulb should be immediately below the side arm so that the vapors must surround the bulb before passing into the condenser1. Place one or two boiling chips in the flask (to promote even boiling and avoid "bumping"). Do not distill until there is no liquid left2. During the distillation, check the distillation rate. If an acceptable distillation rate is not obtained, adjust the variac until an acceptable rate is obtained. After the last fraction is collected, turn the distillation off and let cool before disassembling. Empty all liquids into the appropriate “used” mixture container. For Parts 1, 2 and 3, calculate the percent A, percent I and percent B obtained, based on the initial 50 ml mixture originally used. Calculate the efficiency of the separation for each distillation.

Collection of Pure Fractions: As a general rule of thumb, fractions collected between 5 ºC below the literature boiling point of the pure liquid and 5 ºC above are considered to be pure. On the high temperature end of practical bench distillation, about 130 ºC, the low end is extended by five degrees. At the very limit, about 160 ºC, the low end is extended by ten degrees.

Part 1: Ethanol and 2-Propanol

Use the 100 ml round bottom standard taper flask, matching still head, narrow condenser and adaptor to assemble a distillation apparatus (see sample set-up)4. Add 50 ml of a 50:50 (by volume) mixture of ethanol and 2-propanol (isopropyl alcohol) and distill. Record the "first drop" vapor temperature and the high temperature reached during distillation. Measure and record the volume of the fraction collected. Disassemble the distillation apparatus, placing the fraction collected and the liquid remaining the pot into the “used” ethanol and 2-propanol container.

Part 2: Cyclohexane and Toluene

Using the same apparatus as Part 1, distill 50 ml of a 50:50 (by volume) mixture of cyclohexane(A) and toluene(B). Note that the temperature changes throughout the distillation. Collect three (3) fractions according to your calculated temperature ranges: (A, I, B) and measure the volume of each fraction. The variac setting may need to be increased to keep the rate constant. Record the "first drop" vapor temperature and the high temperature reached for each fraction. To separate all of this mixture into even these impure A and B fractions would require repeated distillations of I (intermediate) fractions. Disassemble the distillation apparatus, placing all the fractions collected and the liquid remaining in the pot in the "used" cyclohexane and toluene container.

Part 3: Acetone and Cyclohexanone

Using the same apparatus as Part 1, distill 50 ml of a 50:50 (by volume) mixture of acetone(A) and cyclohexanone(B). Note that the temperature remains constant while acetone is distilling, then drops. When the temperature drops 5o below your experimental high temperature for Fraction A, change receivers and increase the variac setting by 20. Continue increasing the variac setting by 5 increments every 5 minutes until Fraction B starts to distill. Collect three (3) fractions according to your calculated temperature ranges: (A, I, B) and measure the volume of each fraction. Disassemble the distillation apparatus, placing all the fractions collected and the liquid remaining in the pot in the "used" acetone and cyclohexanone container.

Analysis/Results:

Part 1: Compare the experimental boiling point ranges of the fractions collected and the literature boiling points of the two pure liquids, determine whether any separation was achieved. From the percent recovery of fractions A and B, comment on the efficiency of the distillation.

Parts 2 and 3: Compare the experimental boiling point ranges of the three fractions collected and the literature boiling points of the two pure liquid. Include the volumes collected for the three fractions and determine whether any separation of pure liquids was achieved. From the percent recovery of fractions A and B, comment on the efficiency of this distillation.

In general, comment on the relative efficiency of separation by simple distillation as a function of the difference of the boiling points of the two liquids being distilled. Be sure to include the barometric pressure.

Notes:

1. If a reflux ring is observed in the distillation head, the thermometer should be placed with the top of the bulb just below the reflux ring, instead of just below the side arm.

2. **NEVER HEAT A SYSTEM WITH NO REMAINING LIQUID.** It is not safe to heat a distillation flask when there is no liquid left. Remove the heat just before the flask goes completely dry, usually when the boiling chips become visible through the foam.

3. A reasonable rate of distillation is important - some 25 to 45 drops of condensate per minute is about right. Too great a heat will cause flooding of the still head or additional heating of the vapors: the latter prevents liquid-vapor equilibrium so that the temperature reading is not the boiling point. Too small a heat results in intermittent distillation, with fluctuating and meaningless temperature readings. However, even in the acceptable range speed of distillation is usually traded for lost efficiency. If you have difficulty controlling your variac, ask for help.

4. Be gentle in assembling apparatus with ground glass joints. There is no flexibility. Tighten clamps and holders gently. Before putting ground glass joints together, grease inner member with a light film of "joint grease". Use just enough so that the ground glass loses its frosted appearance: too thick a film will dissolve out and cause leaks.

5. It is safer to attach the rubber tubing to the condenser and to insert the thermometer in the thermometer adapter before assembling the apparatus. The cooling water should flow in the lower end, out the upper end (“in the bottom and out the top”), of the condenser jacket. This sends the warmed water out immediately. Use a gentle flow of water. Before distilling check to see that all the joints in your apparatus are tight: leaking vapors cause fires and loss of substance being distilled. Use clips even if joints are tight.

Calculations:

Determination of the Percent Recovery: For each fraction collected during the distillation the percent recovery is calculated based on the original volume of liquid mixture that was used:

$$\%A = \frac{\text{mL of fraction A recovered}}{\text{mL of original volume A used}}$$

Determination of the Efficiency of a Distillation: The efficiency of a distillation is based on the amounts of pure liquids obtained from the distillation and is calculated using the following formula:

$$\text{Efficiency} = \frac{\%A + \%B}{\%A + \%I + \%B}$$

where A is the low boiling point pure liquid, I is the intermediate, and B is the high boiling point pure liquid.

CJF

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