Rowan College at Burlington County

CHE 241

Lab 1: Simple Distillation

Video Explanation of this lab

What is distillation? Here's a lecture video.
This is the slideshow from the video.

Here is a sample of what a simple distillation looks like.
Here's a video of an actual fractional distillation apparatus.

In this lab you are going to explain how to carry out three distillations. Before you get started, read the lab procedure below. This is taken directly from the lab supplement found here.

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.

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-propane 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.

It is obvious that this lab is asking you to carry out simple distillation based on the experimental set-up. But for this lab, we want you to act as a “Distillation Consultant,” someone who is an expert so that we can effectively separate these three mixtures with as much purity as possible while considering time and energy output.

The company that hired you tried to perform the distillations above in the method described. In order to accurately assess the information they provide, you first must identify the boiling points of each liquid. Look it up and fill out the table below with the information.

Compound	Boiling Point (℃)	Reference Source
Ethanol		
2-Propanol		
Cyclohexane		
Toluene		
Acetone		
Cyclohexanone		

Now, looking at the three distillations, make an initial assessment of each one. In your assessment, state explicitly if you think the simple distillation process worked. Also include an explanation of why you think the way you do.

Part 1 Assessment:

Part 2 Assessment:

Part 3 Assessment:

Now that the initial assessments are complete, it's time to get into the nitty gritty of consultancy. Let's help your customer out!

Let's start with the "Part 3" distillation first.

The experiment was carried out by the customer and the following results were determined for their distillation:

	Trial 1 Composition (%)	Trial 2 Composition (%)	Trial 3 Composition (%)	Trial 4 Composition (%)
Acetone	46	51	50	50
Intermediate	2	3	2	--
Cyclohexanone	50	46	47	49

Efficiency:

Average Efficiency:

For each trial, calculate the efficiency and then determine the average efficiency for all trials. The formula for efficiency is below, taken from the lab handout.

> **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:
>
> Efficiency = (%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.

Make a judgement call. Was this distillation successful? If so, how do you know? If not, explain what went wrong.

Let's move to "Part 2" distillation.

The experiment was carried out by the customer and the following results were determined for their distillation:

	Trial 1 Composition (%)	Trial 2 Composition (%)	Trial 3 Composition (%)	Trial 4 Composition (%)
Cyclohexane	38	43	42	40
Intermediate	20	21	24	20
Toluene	40	35	33	38

Efficiency:

Average Efficiency:

For each trial, calculate the efficiency and then determine the average efficiency for all trials.

Make a judgement call. Was this distillation successful? If so, how do you know? If not, explain what went wrong.

And finally, let's look at "Part 1" distillation.

The experiment was carried out by the customer and the following results were determined for their distillation:

	Trial 1 Composition (%)	Trial 2 Composition (%)	Trial 3 Composition (%)	Trial 4 Composition (%)
Ethanol	2	3	0	3
Intermediate	90	88	98	92
2-Propanol	0	1	0	2

Efficiency:

Average Efficiency:

For each trial, calculate the efficiency and then determine the average efficiency for all trials.

Make a judgement call. Was this distillation successful? If so, how do you know? If not, explain what went wrong.

Now for some analysis and recommendations.

Take a few minutes to look at the average efficiency of each trial. Compare the average efficiency to the difference in boiling point (ΔBP) of the two components in the mixture. Identify a relationship between efficiency and ΔBP.

If the target is to get close to 100% efficiency, how does the relationship above help you make a decision about which type of distillation to use?

For part 1, recommend which type of distillation to use. You must choose from simple, fractional, and vacuum distillations. Explain your choice in detail using data from the lab.

For part 2, recommend which type of distillation to use. You must choose from simple, fractional, and vacuum distillations. Explain your choice in detail using data from the lab.

For part 3, recommend which type of distillation to use. You must choose from simple, fractional, and vacuum distillations. Explain your choice in detail using data from the lab.