Organic Chemistry Experiment 2: Purifying Chemicals by Distillation

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Lab Section: Tuesday Mornings (10:00-13:00), Odd Weeks

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Procedure

Part A. The Simple Distillation

1. The simple distillation apparatus was removed from the locker and assembled in the fume hood. The assembling of the glassware and apparatus is as described in the lab manual (CHM 1321 Introductory Organic Chemistry Laboratory Manual 2017, Dr. William Ogilvie and Dr. Tony Durst, 2017, Exp.1, p. 20 to 27).
2. A 100mL distillation flask was used as the distilling flask and a 10mL graduated cylinder was used as the receiving flask. The apparatus was secured by being clamped to metal rods in the fume hood.
3. A magnetic stir stick was placed into the distilling flask and then the flask was inserted into a heating mantle. The heating mantle rested on a magnetic stir plate.
4. 50 mL of a 50:50 mixture solution of 2-propanol and 1-butanol was put into the distillation flask via a long stem glass funnel.
5. A water input hose was attached to the lower segment of the condenser while a water output hose was attached to the upper portion of the condenser to direct the flow of water up and out of the condenser. The condenser was then attached to the top of the distilling flask.
6. A thermometer was put into the distillation head such that the tip was below where the condenser was attached.
7. The magnetic stir plate and heating mantle were plugged in. The heating mantle was given 120 V (80%).
8. Once the mantle was heated and the magnetic stir stick was stirring the solution at a steady rate at the centre of the mixture, the water for the condenser was turned on by turning a knob on the side of the fume hood.
9. The solution was distilled while the temperature was noted at 1mL intervals of distillate received in the 10mL graduated cylinder (receiving flask). These values were written in a raw data table.
10. The distillation was allowed to proceed until 20mL of distillate was produced.

Part B. The Fractional Distillation

1. The apparatus and flasks were allowed to cool down between distillations.
2. The distillate of 50 mL of 50:50 mixture solution of 2-propanol and 1-butanol from the simple distillation was reused in the fractional distillation portion of the experiment. It was reinserted into the distilling flask of the apparatus. The magnetic stir stick remained in the distilling flask.
3. The fractional distillation apparatus was assembled in the fume hood by using the metal rods in the fume hood, some glassware from the simple distillation, and a fractionating column obtained from the lab demonstrator.
4. A fractionating column densely packed with mental sponge was attached to the distillation head. A 100mL distilling flask and a 10mL graduated cylinder (receiving flasks) were used again in this distillation.
5. The thermometer was inserted into the distillation head such that the tip was below where the condenser was attached.
6. The water input and output hoses and a condenser were attached to the apparatus, and the water, magnetic stirring plate and heated mantle were turned on in the same way as the simple distillation.
7. Once the heating mantle was heated and the stir stick was stirring the solution at a steady rate, the fractional distillation proceeded.
8. The temperature was noted at 1mL intervals of distillate received in the 10mL graduated cylinder (receiving flask). These values were written in a raw data table.

11. The distillation was allowed to proceed until 20mL of distillate was produced.

Observations

Part A. The Simple Distillation

- The 50:50 mixture solution of 2-propanol and 1-butanol was a colourless and transparent liquid with an odour like the scent of a permanent marker.
- The temperature increased steadily (linearly) during the distillation.
- Multiple air bubbles appeared in the condenser and cold condensation was noted on the side of the glass tube.
- The distillate had the same appearance and smell as the solution before the distillation (it was still a colourless and transparent liquid with a bitter odour).

Part B. The Fractional Distillation

- Distillation proceeded at a continual and steady rate, until 10mL of distillate was produced. At this point, the temperature jumped up approximately 10°C in the 1mL interval.
- The fractionating column was packed with thin strands of metal that were lustrous and silver in colour.
- The compound with the higher boiling point, in this case the 1-butanol, dripped back down the fractionating column to the distilling flask, therefore a substance was noted on the side of the tube and droplets of the substance were observed around the metal strands.
- Multiple air bubbles appeared in the condenser and cold condensation was noted on the side of the glass tube.

Results

Table 1. Temperature of the Distillate Recorded Every 1 mL Increment During the Simple Distillation

Volume of Distillate (mL)	**Temperature (°C)**
1	85.6
2	87.0
3	87.8
4	88.7
5	89.5
6	90.3
7	91.5
8	92.8
9	94.3
10	95.5
11	97.0
12	99.9
13	102.9
14	106.0
15	109.4
16	112.3
17	114.9
18	116.3
19	117.2
20	117.6

Table 2. Temperature of the Distillate Recorded Every 1 mL Increment During the Fractional Distillation

Volume of Distillate (mL)	**Temperature (°C)**
1	82.0
2	82.5
3	83.2
4	84.0
5	84.8
6	85.6
7	86.1
8	87.6
9	89.8
10	93.0
11	97.6
12	107.0
13	112.1
14	115.4
15	116.4
16	116.9
17	117.0
18	117.0
19	117.0
20	117.0

Figure 2. Temperature (°C) vs. Volume of Distillate (mL) for the Simple Distillation

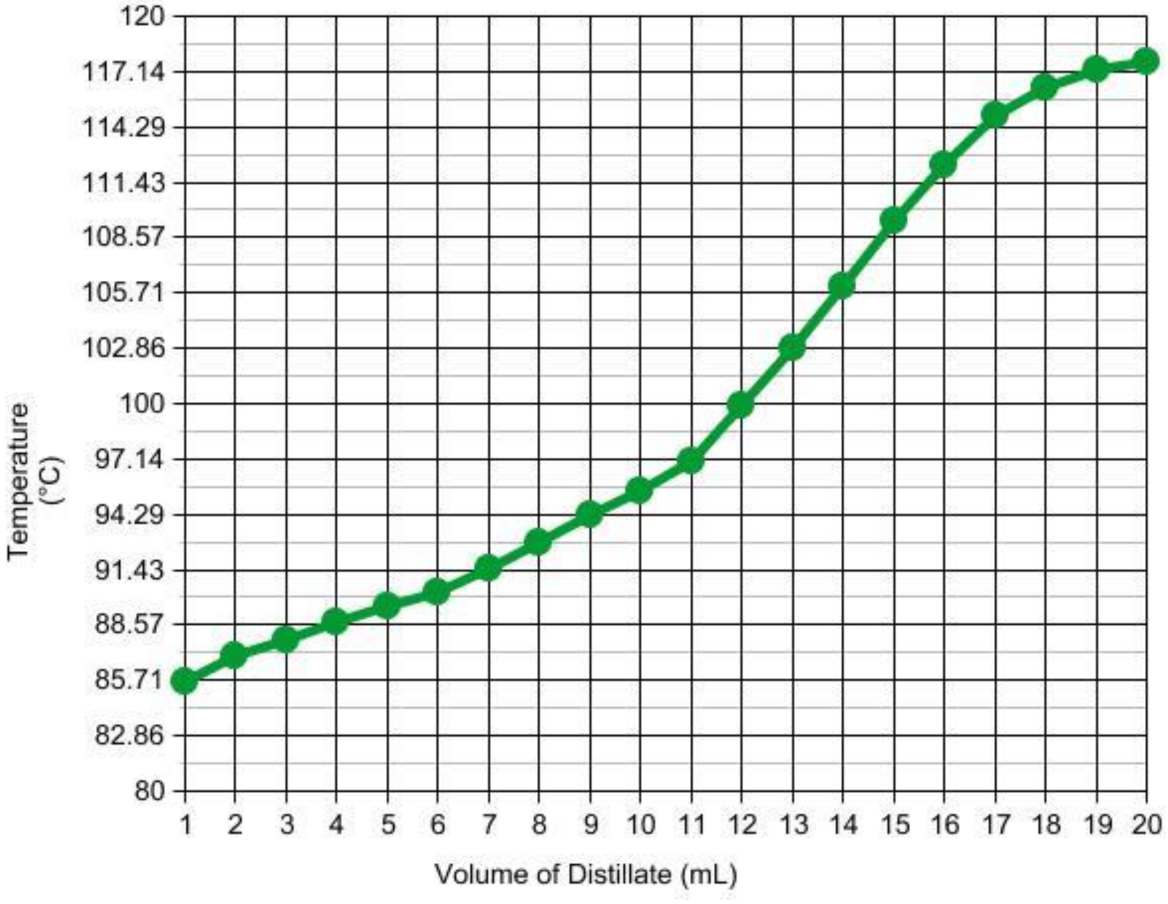
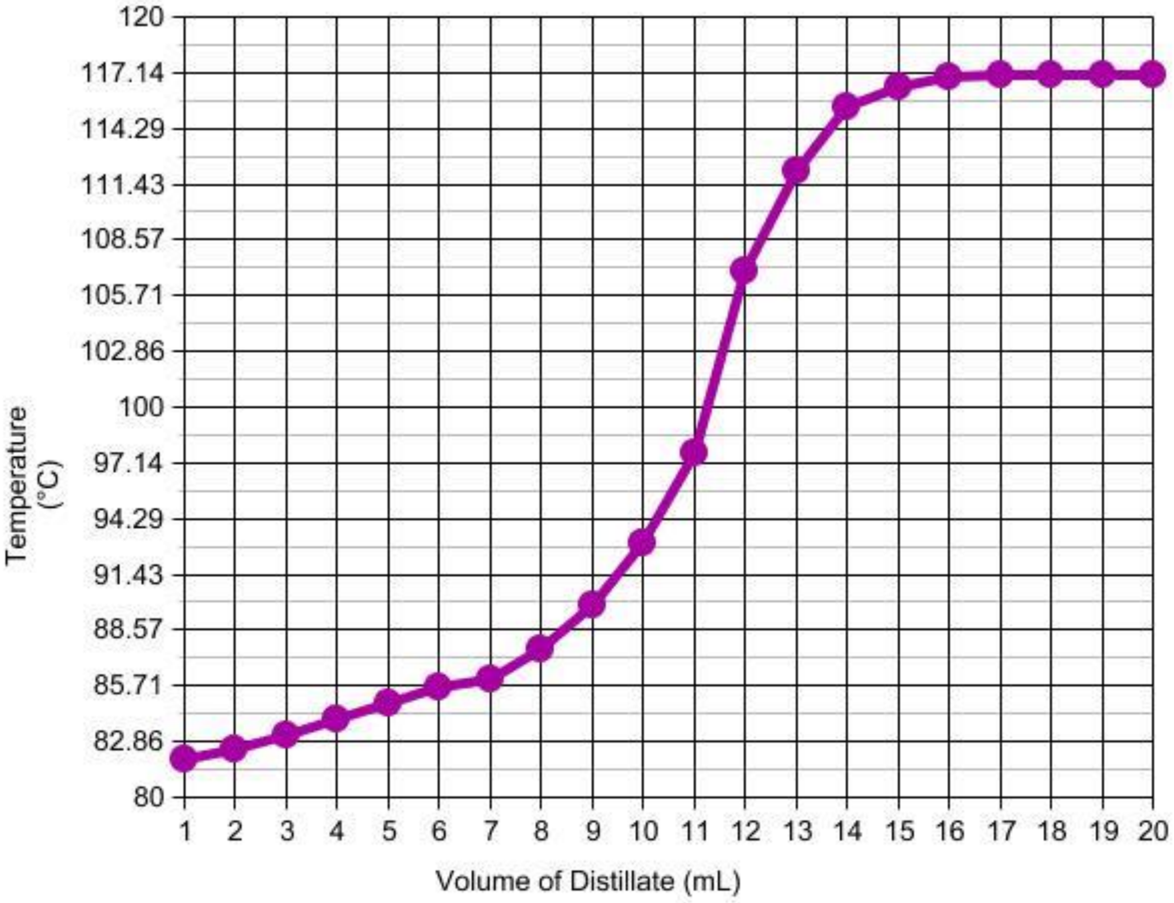


Figure 3. Temperature (°C) vs. Volume of Distillate (mL) for the Fractional Distillation



Discussion

The simple distillation resulted in a less effective separation of the components of the 2-propanol to 1-butanol solution as compared to the fractional distillation of the same mixture of substances. The two distillation mechanisms differed only by the addition of a fractionating column packed with lustrous silver-coloured metal in the fractional distillation. The mechanism of the simple distillation used only a distilling head directly attached to the distilling flask; this meant there was a relatively short length and area over which separation could occur. The mechanism of the fractional distillation used a long glass tube packed with many strands of lustrous silver-coloured metal. The fractionating column was put between the distilling flask and the distilling head; this provided a greater length and area over which the vapours of the substance traveled before reaching the condenser and condensing back down into liquid and going into the receiving flask. This fractionating column was tightly packed with metal strands which increased the surface area and regions upon which the vapours could condense and further separation could occur. Also, some liquid caught on the metal sponge. This fractional distillation allowed for multiple distillations to occur on the metal sponge packed into the fractionating column. The surface area of the fractionating column increased with the input of the metal sponge, which increased the number of surfaces where the substance's vapours could catch onto, condense into liquid once again and then re-evaporate. This condensation and re-evaporation of the vapours in the long glass metal-packed tube resulted in a better separation of the compounds because many individual distillations occurred, whereas in the simple distillation, only one single distillation could occur.

The fractional distillation achieved a higher product purity compared to the simple distillation, since the fractional distillation uses a process of multiple re-distillations. Even though this process is less efficient since it requires a greater amount of time to do the multiple distillations, it is a more effective process than the simple distillation.

Also, it is important to note that the composition of the starting 50:50 mixture solution of 2-propanol and 1-butanol perhaps varied a bit between the simple and fractional distillation due to the separation of the compounds a first time in the simple distillation and then the distillate was poured back into the distilling flask to be used again for the fractional distillation. However, the thorough mixing of the solution in the distilling flask between distillations by the magnetic sit stick ensured that the component ratio of the starting mixture was basically the same at the start of both distillations.

Separation in composition of the substance occurred as a smaller fraction of the compound with the lower boiling point continued up the column as vapour, while a larger fraction of the compound with the higher boiling point remained as liquid and fell back into the distilling flask. Vapour is continually released from the heated mixture in the distilling flask and then travels up the fractionating column where it cools, condenses to liquid droplets on the metal strands. Then, it returns to vapour in the column as heat from the distilling flask seated on the heating mantle is radiated up into the column. When it is turned back into vapour, it can finally continue into the condenser and travel to its final destination in the receiving flask.

A larger fraction of the compound with the lower boiling point is present in the vapour that escapes from the heated mixture, since Raoult’s Law conveys that the vapour pressure of the compound with the lower boiling point is greater than that of the compound with the higher boiling point. Therefore, the composition of the vapour contains a greater amount of the compound with the lower boiling point. Upon condensation, the droplet retained its ratio of a greater amount of the compound with the lower boiling point and less of the compound with the higher boiling point. However, a fraction of the droplet was vaporized again due to the subsequent vapors and radiated heat. The fraction of the droplet containing the compound with the higher boiling point does not get enough energy to become vapour to it remains as a liquid on the metal, whereas the compound with the lower boiling point does obtain the energy it needs to become vapour. As a result, the compound with the higher boiling point falls back down into the distilling flask as a droplet of liquid and the compound with the lower boiling point continues on as vapour. This separation process occurs multiple times as the vapours continue to travel upwards, each time condensing with the lesser fraction of the droplet favouring the compound with the lower boiling point, evaporating it and rising it, and the compound with the higher boiling point cannot remain as vapour and drips back down as liquid.

The temperature at which the vapours escape from solution rises as the mixture increases in concentration of the compound with the higher boiling point. As the distillation proceeds, the vapours that reach the top of the fractionating column start to consist mostly of the compound with the lower boiling point, while the mixture in the flask consists mostly of the compound with the higher boiling point, whose evaporation would begin to occur if the temperature rises enough. In this experiment, the rising vapours exhibits a higher concentration of the compound with the lower boiling point, which in this case is the compound 2-propanol, while the solution in the flask accumulates a greater amount of the compound with the higher boiling point, which was the compound 1-butanol. The temperature at which 1butanol vaporizes exceeds the temperature at which 2propanol vaporizes and thus as the vapours increased in composition of 2-propanol and decreased in composition of 1-butanol.

The ‘Temperature vs. Volume of Distillate’ graph for the simple distillation depicted a relatively linear curve that appeared to increase at a constant rate; the temperature rose consistently over the 1 mL intervals. However, there was a slight increase in the rate of temperature increase over the interval of 11-17mL. The constant increase in temperature between the 1-10mL intervals indicated that the vapour contained only the compound with the lower boiling point, 2-propanol. The slight rise in vapour temperature indicates the point at which the compound with the higher boiling point, which was the compound 1-butanol, started to vaporize as well, as the compound with the lower boiling point compound, 2-propanol, had been mostly extracted from the mixture by vaporization. The steadying out of the curve one again after 10mL point means that the vapour contained a combination of both compounds which favored both boiling points of the two compounds, meaning both receive enough heat energy to turn to vapour. As the boiling point of 2-propanol was 82.6 ºC and the boiling point of 1-butanol was 117.7ºC, the vapours exhibited a temperature within these parameters. As the temperature rose, the component ratio of the vapours favoured 2-propanol, but full separation was not complete, as the vapours rising from solution contained fraction of 1-butanol as well. Although this fraction slightly decreased over time, full separation was not ensured, as the component ratio of the vapour shifted throughout the distillation. It was expected that the simple distillation temperature vs. volume of distillate would result in a smooth, linearly-increasing curve as the temperature increases at a linear rate as continual shifting of the component ratio of the vapours occurs, but experimentally, the curve was not perfectly linear.

The ‘Temperature vs. Volume of Distillate’ graph for the fractional distillation had a more pronounced rise in the curvature and it was over the 9-14mL interval. The curve increases initially at a constant rate and then begins to increase more rapidly from when the volume of distillate was 9 mL to 14 mL. At the beginning of the distillation, the temperature increased in approximately 1ºC increments as the compound with the lower boiling point, the compound 2-propanol, was vaporizing out of the solution in the distilling flask. The start of the more pronounced rise in temperature indicated the point at which 2-propanol had nearly completely evaporated from the 50:50 mixture solution. At this point, the 1-butanol compound with the higher boiling began to be extracted from the solution. The distillation was terminated when the temperature reached the boiling point of the 1-butanol, depicted by the flattening of the curve and that the temperature past 16ml of distillate remained at 117ºC. This slightly more rapidly increasing temperature curve for the fractional distillation was expected, as the multiple distillations allow for the compounds to be separated more effectively. This results in the vaporization of one of the compounds at its boiling point before the vaporization of other compound and then the other compound with the higher boiling point is then vaporized as the temperature reaches the higher boiling point value. The more rapidly increasing temperature (the sharp rise in curvature) depicts the transition between the two boiling points.

Possible sources of error include the fact that my lab partner and I used a 10ml graduated cylinder as the receiving flask, but the volume of distillate that we collected was 20ml; this meant that we had to quickly switch out the filled graduated cylinder at 10ml of collected distillate with an empty one. This could mean that the volume of distillate collected could have been a little less or more than exactly 20ml. However, we ensured that we switched the cylinders quick enough so that no distillate dripped out just onto the counter (the cylinders caught all the droplets that fell). Also, another error could have occurred in transferring the mixture back into the distilling flask for the fractional distillation. It was not possible to ensure that all 20 mL of the solution gathered in the graduated cylinder (receiving flask) was transferred, since a small amount of the mixture may have clung to the sides of the glassware or to the inside of the apparatus. Also, slight human errors in recording the temperatures may have occurred as the time at which the 1 mL of distillate was noted, as the temperature is ever-increasing and it takes time to look over to the thermometer after the distillate reaches another milliliter increment. A mechanism that could monitor both the volume in the receiving flask and the temperature shown on the thermometer at the same time instantaneously would reduce the margin for human error. Additionally, determining the density of the distillate substance after the distillation could further tell us the purity of the extracted compound, in this case the compound 2-propanol.

Questions

1. During the fractional distillation, we have the liquid flow back through the fractionating column so that effective separation of the components of the 50:50 mixture of 2-propanol and 1-butanol can occur. The droplets of liquid that drip back down the column consist mainly of the compound with the higher boiling point (the 2-propanol), which cannot retain enough energy to remain vaporized. It doesn't make it all the way up the column and subsequently into the condenser – it turns back into liquid in the column or on the metal strands, and then returns to the distilling flask, since the heat is not high enough to keep it as a vapour (its boiling point temperature has not been reached). Otherwise, the absence of the falling droplets would indicate that the mixture has been overheated and complete vaporization of the both compounds has occurred (a temperature that caters to both compound's boiling points and therefore allows for both compounds to remain as vapour has been reached).

2. A uniform temperature gradient in the fractionating column would allow more distillations to occur. If the temperature in the column suddenly dropped, the decreased temperature would cause the vapours would diminish the heat of the rising vapours, therefore they would condense back down to liquid and return to the mixture in the distilling flask without being slightly reheated for partial vaporization in the column. So, a smooth temperature gradient is favourable so that a constant state of vapour condensation and evaporation occurs in the metal-packed fractionating column. Also, if the temperature fluctuates between the correct temperature and a higher temperature, some of the compound with the higher boiling point may start to vaporize and be distilled on and off, along with the compound with the lower boiling point; this would cause the distillate purity to be less and separation of the compounds would be flawed. In the fractionating column, the temperature decreases towards the top as the purified and separated vapours then enter the condenser where they are condensed and reach the receiving flask.

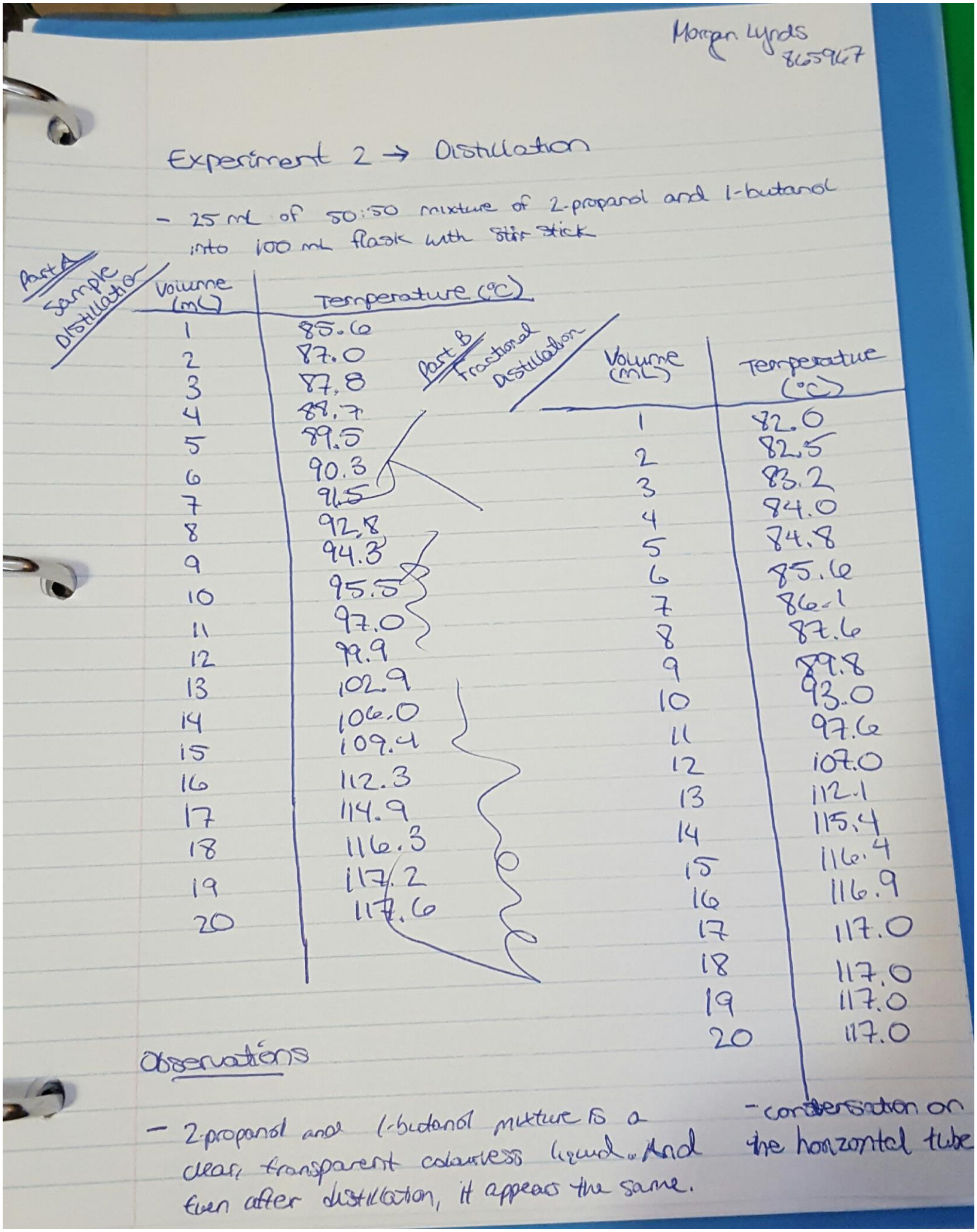
3. The boiling point of a liquid is defined as when the vapour pressure exerted by the liquid is equal to the atmospheric pressure. Therefore, when the temperature of benzene reaches its boiling point of 81°C, the vapour pressure of benzene would be equal to the atmospheric pressure, which is 1.0 atm (760 mmHg or 101.325 kPa).

4. An increase in the atmospheric pressure would increase the boiling point of a liquid. The liquid would be forced to exert a higher vapour pressure to counteract the greater atmospheric pressure that is pushing down on the liquid. A greater amount of energy in the form of heat would be required by the higher vapour pressure. Therefore, increasing the vapour pressure so that it equals the increased atmospheric pressure would raise the temperature at which the liquid boils.

5. A more complete filling of the condenser's water jacket is ensured by inputting the cooling water at the bottom of the condenser. If the cooling water was inputted at the top if the condenser, it would not get filled at fully and the water would flow down and disperse into streams as it flowed to the bottom of the condenser; gravity would force the water down the condenser and the attraction would cause the water to unevenly distribute in the condenser's water jacket and therefore separate into multiple smaller streams. Also, as the water would attempt to flow out at the bottom of the condenser, air bubbles would form between the water droplets. Therefore, by propelling the water up the condenser from the bottom, a complete and even distribution of water is ensured and forcing water up the condenser ensures that the water evenly fills the space with no air bubbles or a very minimal amount.

6. A 3:1 ratio of A to B in the mixture means that the composition consists of 75% A and 25% B, and Raoult's Law conveys that the total vapour pressure is determined by the partial pressure of pure liquid A multiplied by its mole fraction in the mixture, plus the partial pressure of pure liquid B multiplied by its mole fraction in the mixture. Therefore,
   Vap. pressure of the mixture = (mole fraction A)(vap. pressure A) + (mole fraction B)(vap. pressure B)
   = 0.75 (350 mmHg) + 0.25 (150 mmHg)
   = 300. mmHg

Raw Data



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Experiment 2 → Distillation

- 25 mL of 50:50 mixture of 2-propanol and 1-butanol into 100 mL flask with stir stick

Part A
Sample Distillation

Volume (mL)	Temperature (°C)
1	85.6
2	87.0
3	87.8
4	88.7
5	89.5
6	90.3
7	91.5
8	92.8
9	94.3
10	95.5
11	97.0
12	99.9
13	102.9
14	106.0
15	109.4
16	112.3
17	114.9
18	116.3
19	117.2
20	117.6

Part B
Fractional Distillation

Volume (mL)	Temperature (°C)
1	82.0
2	82.5
3	83.2
4	84.0
5	84.8
6	85.6
7	86.1
8	87.6
9	89.8
10	93.0
11	97.6
12	107.0
13	112.1
14	115.4
15	116.4
16	116.9
17	117.0
18	117.0
19	117.0
20	117.0

Observations

- 2-propanol and 1-butanol mixture is a clear, transparent colourless liquid. And even after distillation, it appears the same.
- condensation on the horizontal tube