Experiment 2: Purifying Chemicals By Distillation

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and

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CHM 1321 Z02

Luana Porto

Protocol:

The procedure was followed as explained from the lab manual for experiment 2:

- Department of Chemistry, University of Ottawa, Experiment 2: Purifying Chemicals by Distillation, September 2014.

Observations:

The solution in the flask was clear, transparent and colorless for both simple and fractional distillation.

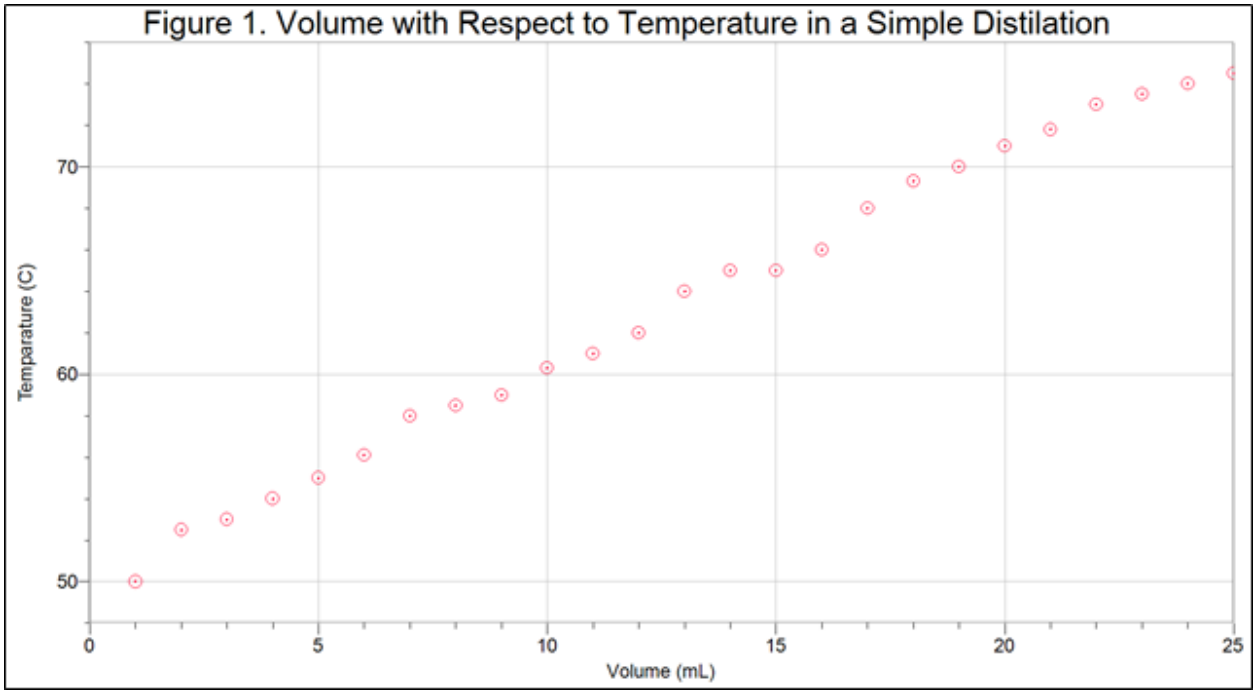
Simple distillation:

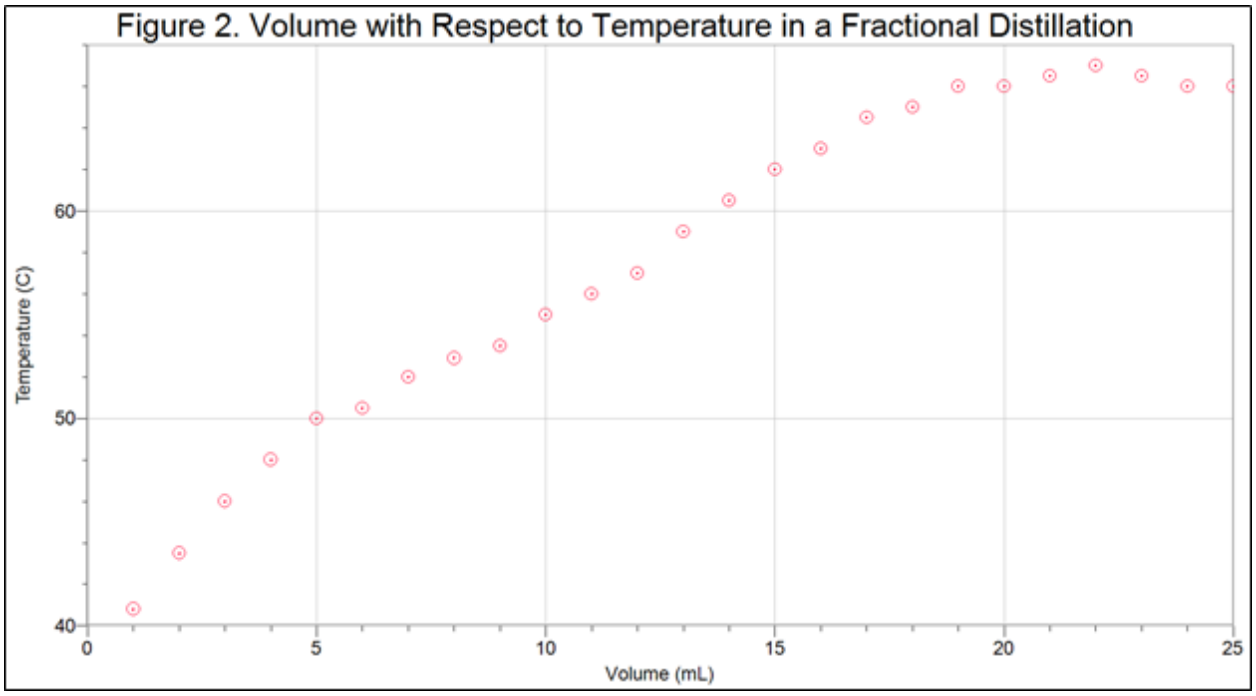
When the experiment started it was observed that there was some condensation in the inside walls of the flask. Secondly as the experiment progressed there was some solution moving down the inside walls of the vertical tube back into the flask.

Fractional distillation:

It was observed that there was solution dripping from the fractionating column back into the flask. Secondly, the graduated cylinder got filled much more slowly by the fractional distillation method compared to the simple distillation method.

Quantitative Data





Discussion:

The solution in the distillation flask is Dichloromethane: Ethyl acetate and is a 50: 50 mixture.

- Dichloromethane has a boiling point of 39.6℃.
- Ethyl acetate has a boiling point of 77.1℃.

Simple distillation:

- The first data point is recorded to be 50℃, which is to be expected. The thermometer measures the temperature by vapor. The minimum boiling point of the solution is 39.6℃ by Dichloromethane; therefore the first recorded temperature must be equal or above this value for the first milliliter of solution distilled.
- Theoretically there should be a plateau at the first boiling point (dichloromethane), then an increase in the graph and then another plateau at the second boiling point (ethyl acetate). There should be a plateau when there is a change in phase from liquid to gas. Heat (energy) is applied to the compound but there is no change in temperature, which causes a plateau. The energy inputted is used to change the intermolecular bonding; in this case bonds are broken causing the compounds to move from liquid to vapor. The heat (energy) is not used for kinetic energy (to increase temperature), but instead the energy is used to change the intermolecular bonding.
- In the graph (Figure 1.) there is no defined plateau for the two boiling points. This can be caused because there is a mixture of the two substances. The vapour at a specific time may contain both compounds which will impact the graph.
- Simple distillation works best if there is only one pure substance being distilled (since there is no interference from another compound). If there is one compound being distilled one horizontal line will be defined on the graph.
- Simple distillation would work better if the two compounds have a very big difference in boiling point, because the vapor would be less mixed at a given time. In the solution provided the boiling point difference is 37.5 ℃.

Fractional distillation:

- The plateaus are much more defined in the fractional distillation then in the simple distillation which is expected. The first plateau in the graph (Figure 2.) is at about 51 ℃ and the second plateau is at 68 ℃.
- The plateaus are much better defined because the two components are separated much better by the fractionating column. Initially there is a lot of dichloromethane vapor. The ethyl acetate is condensed by the fractionating tube back into the distillation flask. This makes a more pure vapor containing mostly dichloromethane initially.
- As the temperature increases the vapor will contain more of the ethyl acetate and at this time most of the dichloromethane would have been distilled. This makes a more pure vapor of ethyl acetate towards the end of the experiment.

- During the lab it was observed that the fractionating distillation took longer than the simple distillation. This makes sense since the fractionating tube caused some resistance and continually condensed vapor ethyl acetate back into liquid.

Sources of Error:

- A temperature plateau did not occur during the simple distillation and may have been the resultant of the changing of the temperature on the heater. The temperature was originally set at 75℃ and later changed to 65℃ which is below the boiling point of ethyl acetate. This may have prevented the distillation from reaching a temperature plateau.
- After the simple distillation finished, the distillation flask was detached from the system and the mixture was spilled. As a result a new batch of the mixture was obtained, which could cause inconsistency as the purity of the mixtures could differ.
- One major source of error was not insulating the joints. In the experiment all joints were connected by using metal clamps and therefore the joints were not air tight. The vapor which could have escaped, could cause heat loss and therefore give lower temperature data.

Conclusion

In conclusion the experiment went as expected for both simple and fractional distillation. The graphical results for the fractional distillation resembled the theoretical graph much more then the simple distillation. This is expected since the fractional distillation produced a better vapor separation.

In the fractional distillation the two plateaus for each boiling point could be observed in the graph (Figure 2.) which is similar to the theory.

Questions:

1. The mixture of ethanol and water is known to be a very good azeotrope, due to the difference of pure boiling points verses the boiling point of the mixture. The boiling point of ethanol is 78.3℃ while water has a boiling point of 100℃. With a 95.6% ethanol and 4.4% water mixture the water boils at 78.1℃. Toluene has a slightly higher boiling point in purity than water does (110℃). Although the chemical properties of the two substances differ largely, the higher the boiling point differs between two substances in a mixture, the better the separation during distillation. Therefore ethanol and toluene would be separable via distillation.

2. If separation is to be precise, a uniform temperature gradient must be maintained in the fractionating column. This is due to the fact that the temperature gradient is a control, and the precision of the mixtures separation is based upon this control. A slight increase in temperature could speed up the separation of the mixture, as the substance with the lower boiling point would

collect in the receiving flask faster than normal. A slight decrease in temperature, and the separating substance will require more heat, and will likely be less pure as it distills.

3. The boiling point of a substance occurs when the vapour pressure is equal to that of the atmosphere acting on it. Assuming that the dichloromethane is boiling under 1 atm, the vapour pressure of dichloromethane at 39.6℃ is 1 atm.

4. Pressure is a factor that determines the energy that molecules in a gas or vapour possess. An increase in pressure causes molecules to pack together in a tighter fashion, increasing movement, vibration, collisions, and therefore; kinetic energy. In other words, increasing the pressure will increase the energy, directly increasing the boiling point. Lowering the pressure also lowers the boiling point of a substance. The two variables are proportional.

5. It is important to have water flowing from bottom to top in the condenser because it ensures the condenser is completely full with water throughout the distillation. As the water climbs against gravity it is slowed, allowing water to enter the condenser at a faster rate than it is leaving. Water must completely fill the condenser to make sure that the vapour leaving the distillation flask is completely cooled and condenses in the receiving flask.

6.

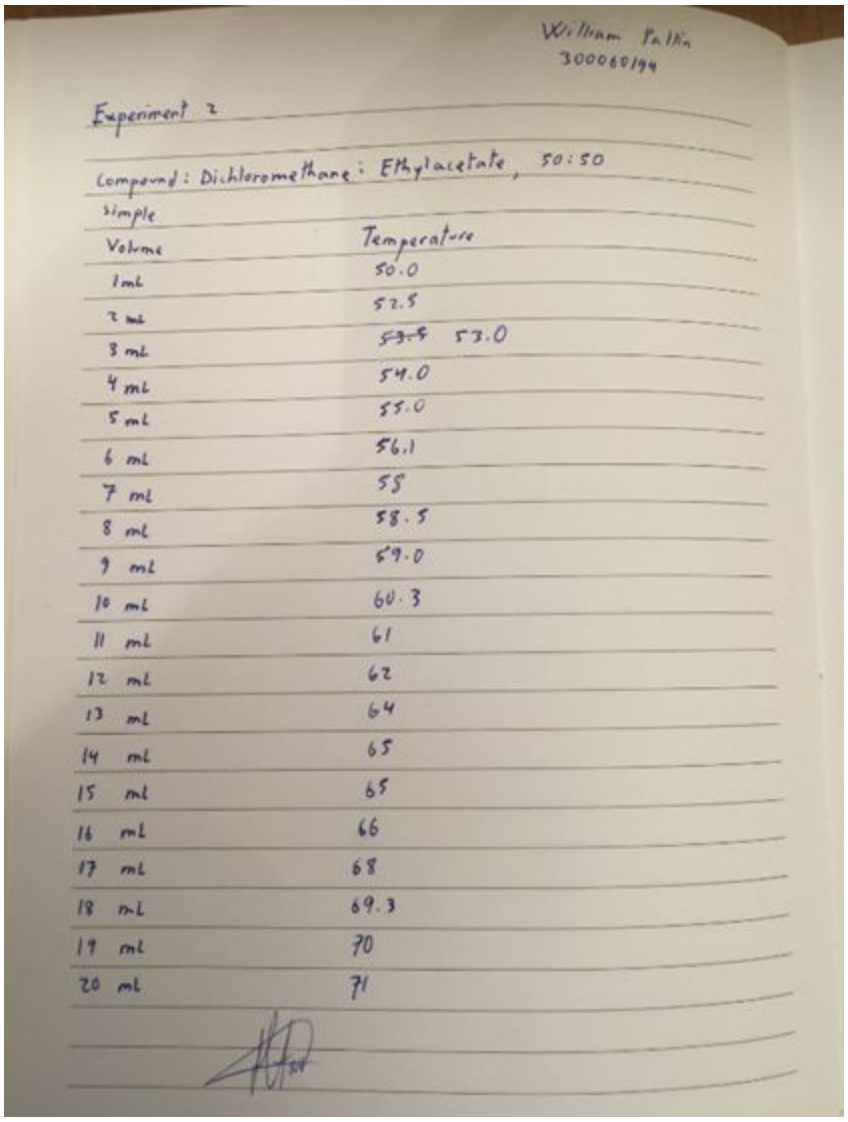
- Mole fraction of A= 4/(1+4) = 0.8
- Mole fraction of B= (1 - 0.8) = 0.2
- Partial pressure A = (mole fraction of A) x (vapour pressure of A)
- Partial pressure B = (mole fraction of B) x (vapour pressure of B)
- P_A = (0.8) x (350mmHg) = 280mmHg
- P_B = (0.2) x (140mmHg) = 28mmHg
- Vapour pressure = (280mmHg) + (28mmHg) = 308mmHg

Therefore the vapour pressure of a 4:1 mixture of A and B at 95℃ is 308mmHg.

References:

- Guggenheim, E.A. The theoretical basics of Raoult's law. Transactions of the Faraday Society, 33 page 151 to 156.
- William Ogilvie, Nathan Ackroyd, C. Scott Browning, Ghislain Deslongchamps, Felix Lee, Effie Sauer, Organic Chemistry Mechanistic Patterns, Chapter 1 and 3, Nelson Education, 2018.

Raw Data



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Experiment 2

Compound: Dichloromethane: Ethylacetate, 50:50

Simple

Volume	Temperature
1 mL	50.0
2 mL	52.5
3 mL	~~53.5~~ 53.0
4 mL	54.0
5 mL	55.0
6 mL	56.1
7 mL	55
8 mL	58.5
9 mL	59.0
10 mL	60.3
11 mL	61
12 mL	62
13 mL	64
14 mL	65
15 mL	65
16 mL	66
17 mL	68
18 mL	69.3
19 mL	70
20 mL	71

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Simple Volume	Temperature
21 mL	71.8
22 mL	73
23 mL	~~74~~ 73.5
24 mL	~~74~~ 74
25 mL	~~73~~ 74.5

Observations

The compound in the flask is clear, transparent and odorless. Their is some condensation in the sides of the flask. Their is liquid going down the sides of the vertical tube. The graduated cylinder is getting filled much slower then it's from simple distillation.

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Fractional distillation

Volume	Temperature
1 mL	40.8
2 mL	43.5
3 mL	46.0
4 mL	48.0
5 mL	50.0
6 mL	50.5
7 mL	52.0
8 mL	~~47~~ 52.9
9 mL	53.5
10 mL	55.0
11 mL	56.0
12 mL	57.0
13 mL	59.0
14 mL	60.5
15 mL	62.0
16 mL	63.0
17 mL	64.5
18 mL	65.0
19 mL	66.0
20 mL	66.0

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Fractional Distillation

Volume	Temperature
21 mL	66.5
22 mL	~~66.5~~ 67
23 mL	~~67~~ 66.5
24 mL	66
25 mL	66

Observations

~~Their are droplets~~ On the sides of the flasks their is ~~tube~~ liquid condensate. Their is liquid dripping from the fractionating column into the flask.

COURSE: CHM 1312 Section Z02 **TA Name:** Luana Porto
YOUR NAME (PRINT): William Paltin **SIGNATURE:** _____

CONFIDENTIAL PEER EVALUATION FORM FOR EXPERIMENT 2

Each team member must submit one assessment form evaluating each **other** member of the team.
Teams may consist of 2-3 members for reports and up to 18 for planning sessions.

You may edit this form.

Do not share or discuss the contents or possible contents of this assessment with others.

In assessing the work of your fellow team members, consider the following aspects:

- Quality of work
- Contribution to the work as a whole
- Ability to get along with others
- Improvements when asked to correct

Team member name	Comments	Grade
Aidan Keller	Honest work, completed questions and sources of error.	5

A – Excellent (5) **B: Great (4)** **C: Good (3)** **D: Fair(2)** **F: Poor (1)**

Note: Do not evaluate yourself on this form

COURSE: CHM 1312 Section Z02 **TA Name:** Luana Porto
YOUR NAME (PRINT): Aidan Keller **SIGNATURE:** _____

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- Ability to get along with others
- Improvements when asked to correct

Team member name	Comments	Grade
William Paltin	Excellent work, completed observations, graphs, and most of discussion.	5

A – Excellent (5) **B: Great (4)** **C: Good (3)** **D: Fair(2)** **F: Poor (1)**

Note: Do not evaluate yourself on this form