Experiment 2

Purifying Chemicals by Distillation

CHM1321-B05
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Joshua Thomas
Student Number: 8572083
TA: Sojung Lee

Procedure:

Here is the link to the procedure which can be found on pages 26 through 27 of the outline.

Citation for Procedure:

Dr. Bianca van Lierop, Dr. Rashmi Venkateswaran, Mr. Alex Bush " Introductory Organic Chemistry", Introductory Organic Chemistry Laboratory Manual, 2017, Experiment 1, p.26-27: https://uottawa.blackboard.com/bbcswebdav/pid-1473670-dt-content-rid-20348841_1/courses/2171_3934/CHM%201321%20Lab%20Manual%202017rvbianca.pdf

Their were two notable modification to the procedure. Firstly, we increased of the heat to ensure that we finish the experiment within the constrained time. Secondly, instead of using a receiving flask we used a graduated cylinder to receive the distillation so that we could measure the quantity of solution that was distilled

Observations:

Simple Distillation Observations:

- Around 28°C the temperature of the solution increased rapidly to approximately 80°C
- At the end of the distillation the solution inside the receiving graduated cylinder, the solution became denser to the point where you could see the solution have more drag as it entered the cylinder

Fractional Distillation Observations:

- Similarly, to the simple distillation at approximately a temperature of 23.9°C the solution increased rapidly to approximately 75°C.
- At the end of the distillation the solution inside the receiving graduated cylinder, the solution became denser to the point where you could see the solution have more drag as it entered the cylinder

Tables:

Table 1.1

mL	Temp ◦C
0	23.9
1	
2	85.9
3	86.4
4	87
5	87.5
6	88.1
7	88.9
8	89.3
9	90.6
10	91.9
11	93.5
12	94.4
13	97.4
14	99.8
5	103.3
16	107.7
17	111.7
18	114.6
19	115.8
20	116.3
21	116.7
22	116.8

In this table, we looking at the results of a simple distillation of a 50:50 mixture of 2-propanol and 1-butanol. We see that the max temperature after 22mL is approximately 116.8 ◦C.

Table 1.2

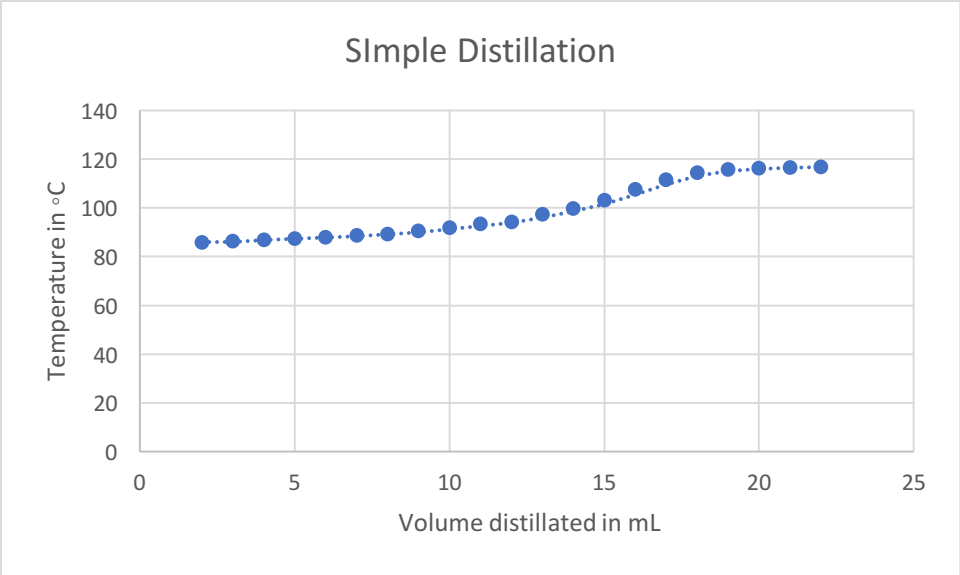
mL	Temp ◦C
0	23.9
1	
2	80.7
3	81
4	81.3
5	81.5
6	81.8
7	82.2
8	83

9	83.7
10	84.2
11	85.8
12	91.1
13	115.5
14	116.7
5	116.9
16	116.9
17	116.9
18	116.9
19	116.9
20	116.9
21	116.9
22	116.9

In this table, we looking at the results of a fractional distillation of a 50:50 mixture of 2-propanol and 1-butanol. We see that the max temperature after 22mL is approximately 116.9 ◦C.

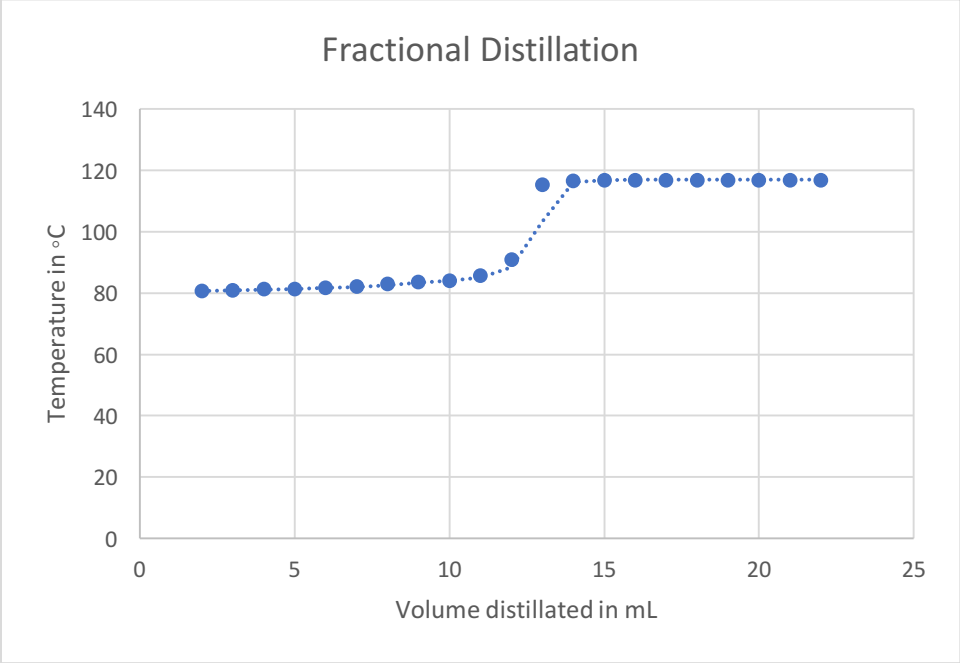
Graphs:

Graph 1.1



This graph represents the simple distillation of the a 50:50 mixture of 2-propanol and 1-butanol. As we can see, the separation of the two solutions is not very evident therefore, an estimation of each solutions volume is not possible.

Graph 1.2



This graph represents the fractional distillation of the a 50:50 mixture of 2-propanol and 1-butanol. As we can see, the separation of the two compounds occurs around approximately 100°C. This also indicates that one of the mixture there is approximately 12mL of one solution and 10mL of the other solution

Discussion:

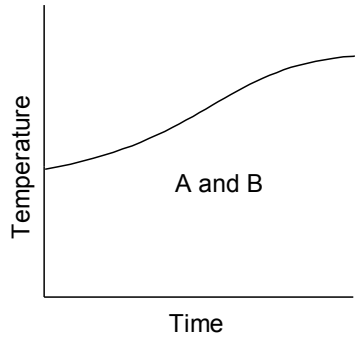
In this section of the laboratory report we will discuss what is distillation and how simple distillation differs from fractional distillation, our results obtained during the experiment, a thorough analysis of our results and graphs and what do they signify and lastly and source of error section with possible improvements to prevent these sources of error.

Firstly, what is distillation? Distillation is a chemical procedure that is widely used to separate a mixture into its respective components by heating up a mixture of different solutions so that they will evaporate at their respective boiling points. [1] This goal is achieved by boiling the mixture which, drives the liquids into their gas phase so that it rises to the top of the distillation column. Then the vapour flows through the condenser where the slow water flow cools the vapour back into its liquid phase. Once the vapour is cooled into its liquid phase it can fall into what's known as a receiving flask. Depending on the efficiency of the separation of the two liquids, this will determine the purity of the sample inside the receiving flask [1]. [2] There are different factors that affect the efficiency of the separation of the mixture using fractional distillation such as: distilling the mixture more than once (known as double distillation), the introduction of packing into the distillation column and the shape of the column. The way packing works is, in a fractional distillation we increase the surface area in the fractionating column by introducing an inert material so that the vapour as it rises, condenses on the inert material in the fractionating

column. Then instead of condensing and going all back to the distilling flask, it lies on the surface of some of the inert material and then once it is evaporating again the vapour will do the same thing at a different height. Using this increased surface area, we can achieve greater separation efficiency of the mixture [2]. Fractional distillation provides a much greater separation efficiency than simple distillation because of its increased surface area which, will be demonstrated when we begin to analyze our collected data during our experiment.

Now that we have some background knowledge of distillation, different types of distillation and how to increase the efficiency of the distillation, we will use this knowledge to help analyze our collected data.

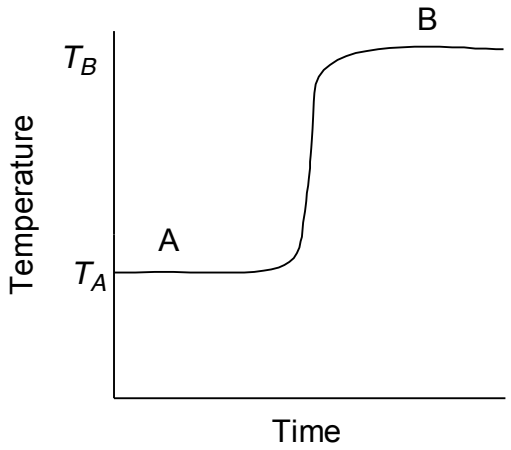
If we examine graph 1.1 which, represents the simple distillation curve we can see, the curve seems to be rather constant as if the mixture of the two solutions was one homogenous solution. We know this is not true. Our mixture in our distilling flask contained a 50:50 mixture of 2-propanol and 1-butanol. The boiling points of both compounds under atmospheric pressure are 82.3°C [3] and 117.6°C [3] respectively. This tells us that the compounds should have evaporated and then condensed into our receiving graduated cylinder at substantially different temperatures which, should have produced two very different segments on our graph. This indicates to us that the separation efficiency of a simple distillation is not very efficient which results in a very pour separation of the mixture into its respective compounds. However, this result is as expected. If we look at the diagram in the lab manual that is inserted below, we can see the gradual increase from compound A to B as if they are one homogenous compound which, is what is shown in graph 1.1 of the simple distillation.



[2]

Moving on. Now if we examine graph 1.2 which, represents the fractional distillation curve we can see, the curve seems to have to distinct lines as if the mixture is composed of two different solutions. We know this is true. Our mixture in our distilling flask contained a 50:50 mixture of 2-propanol and 1-butanol. The boiling points of both compounds under atmospheric pressure are 82.3°C [3] and 117.6°C [4] respectively. This tells us that the compounds have evaporated and condensed into our receiving graduated cylinder at different temperatures which, is demonstrated on graph 1.2. The 2-propanol appears to have begun its distillation at approximately 81°C whereas the 1-butanol seems to have begun its distillation at approximately 116°C. This indicates to us that the separation efficiency of the fractional distillation is much more efficient than the separation efficiency of the simple distillation. The increase of separation efficiency of fractional distillation is a directly caused by the increased surface area inside the fractional distillation column. This result in a greater separation efficiency as mentioned above. If we compare the diagram in the lab manual that is inserted below to our graph 1.2, we can see the

distinct increase from compound A to B because the mixture is composed of two different compound which, is what is shown in graph 1.2 of the fractional distillation.



[2]

Overall, considering our two graphs 1.1 and 1.2, one would be able to agree that our collected data is acceptable because it demonstrates a strong representation of the not very efficient separation of the mixture using simple distillation and it demonstrates strong efficiency of separation for the fractional distillation. We could infer these results because in fractional distillation, the surface area inside the fractionating column is much greater than the surface area inside a simple distillation, which again leads to more precise separation of the mixture into its respective compounds.

For the final part of the discussion section I will discuss some potential sources of error that we may have encountered throughout the experiment.

- One likely source of error that could have affected some results of the experiment could have been inadequately sealed glassware joints as well as possible contaminants in the glassware that were not properly cleaned out from the last groups experiment. If we account for possible contaminants inside the glassware, that could account for our marginal error in the boiling points of our respective solutions.

Questions & Answers:

1. Explain why you must have liquid flowing back through the fractionating column in order to get separation of the components during a fractional distillation.

You must have liquid flowing back through the fractionating column in order to get separation of the components during a fractional distillation because the more times you can evaporate a mixture into its respective components, the purer your sample will be. When you first evaporate some of the mixture out of the distillation flask, it is not 100% pure because it is very likely that it is carrying both compounds in the vapour form. So by flowing the cold water back through the fractionating column, we are condensing again before it is placed in the receiving flask and then once we evaporate it at a certain temperature, the solution now becomes much more pure than it was before. By repeating this process, we will have the purest substance possible.

2. Fractionating columns normally work better if they are insulated in order to maintain a smooth temperature gradient in the column. Why is it important to maintain a uniform temperature gradient in a fractionating column?

Essentially it is the factor that controls the purity of the sample collected in the receiving flask. If the temperature is higher in some spots then others the compound with the higher boiling point will also evaporate with the compound that has the lower boiling point resulting in impurities in the receiving flask, conversely, if some spots are colder than others than the solution which boils at a lower temperature will evaporate and condensate again leading to very minimal solution in the receiving flask.

3. The boiling point of benzene is 81°C. What is the vapor pressure of benzene at this temperature?

The boiling point benzene at 81°C is 1.01325 bar (just use atmospheric pressure)

4. What effect does an increase in atmospheric pressure have on the boiling point of a liquid?

The effects of an increase of atmospheric pressure on the boiling points is directly proportional. Meaning if you increase the pressure, you must increase the boiling point of the liquid as well. This quatity can be measured using Gay-Lussac law which is…

$$\frac{P_1}{T_1} = \frac{P_2}{T_2}$$

Where P_1 is the initial pressure, T_1 is the initial temperature, P_2 is the new pressure and T_2 is the new temperature.

5. Why is it important to have cooling water enter the bottom of the condenser and not the top?

The reasons why the cold water enters from the bottom of the condenser and not the top of the condenser is because if the cold end of the water coming through the condenser is beside the hot vapour that is rising, the condenser could undergo thermal shock and fracture the condenser. In addition it increases the efficiency of the separation

6. Compound A has a vapour pressure of 350 mmHg whereas compound B has a vapour pressure of 150 mmHg at the same temperature. If A and B are miscible, what is the vapour pressure of a 3:1 mixture of A and B at 95°C

Using the 3:1 ratio of mixture we know that the mole fraction of A is 0.75 and that means that the mole fraction of B=1-0.75-0.25. From these, we can use Raoult's Law which says…

$$P_{total} = (P_A * N_A) + (P_B * N_B)$$

Where P_A and P_B are the partial pressured of A and B and N_A and N_B are the mole fracions of each gas.

So…

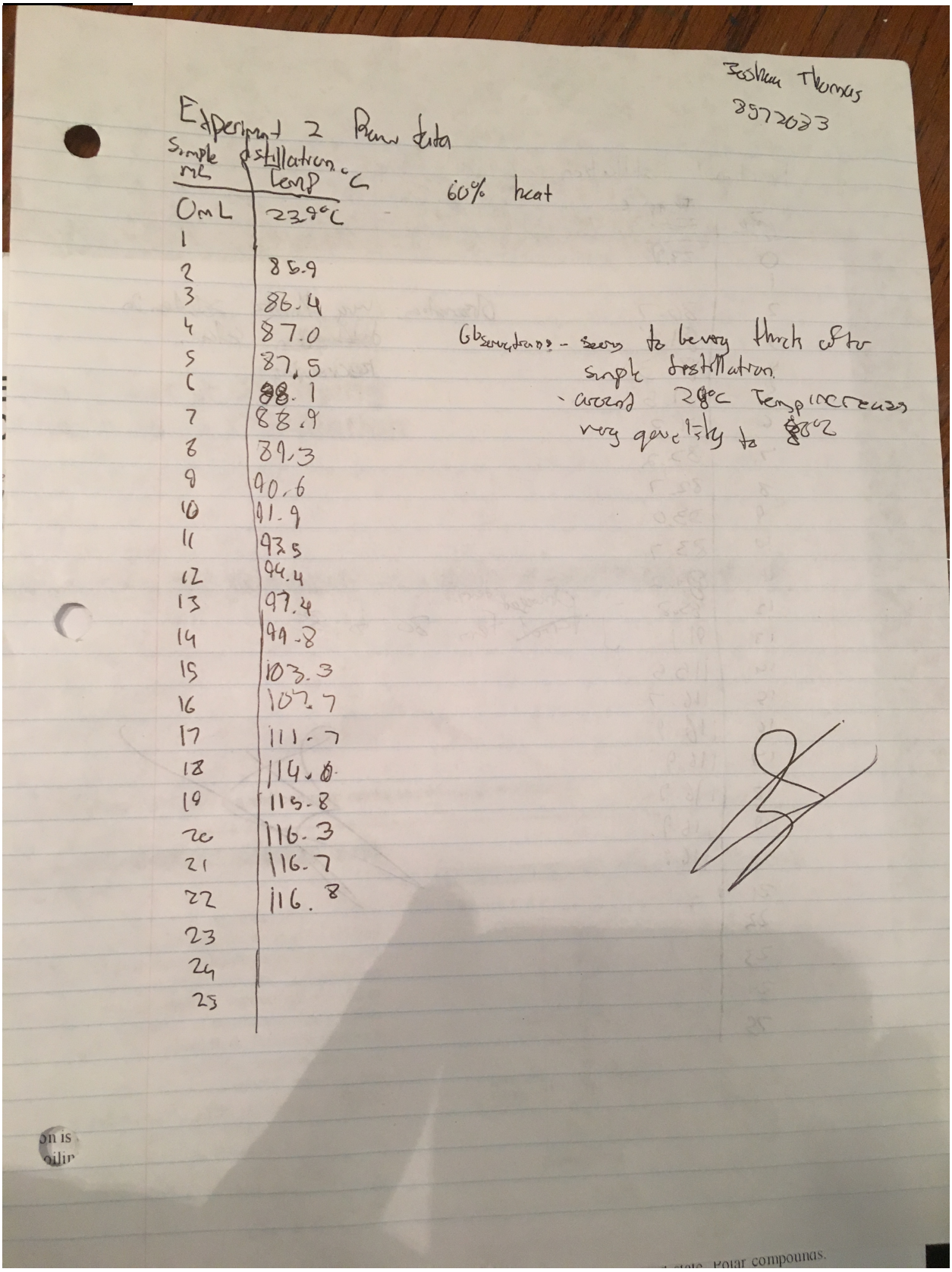
$$P_{total} = (P_A * N_A) + (P_B * N_B)$$
$$P_{total} = (350 * 0.75) + (150 * 0.25)$$
$$P_{total} = 262.5 + 37.5$$
$$P_{total} = 300\ mmHg$$

Therefore, the vapour pressure of a 3:1 mixture of the A and B mixture at 95°C is 300mmHg.

References:

1. H. (2016, August 10). Understand How Distillation Works. Retrieved February 01, 2017, from http://chemistry.about.com/cs/5/f/bldistillation.htm
2. Dr. Bianca van Lierop, Dr. Rashmi Venkateswaran, Mr. Alex Bush " Introductory Organic Chemistry", Introductory Organic Chemistry Laboratory Manual, 2017, Experiment 1, p.20-27: https://uottawa.blackboard.com/bbcswebdav/pid-1473670-dt-content-rid 20348841_1/courses/2171_3934/CHM%201321%20Lab%20Manual%202017rvbianca.pdf
3. Isopropanol | (CH3)2CHOH - PubChem. (n.d.). Retrieved February 01, 2017, from https://pubchem.ncbi.nlm.nih.gov/compound/isopropanol#section=Taste
4. 1-butanol | C4H10O - PubChem. (n.d.). Retrieved February 01, 2017, from https://pubchem.ncbi.nlm.nih.gov/compound/1-butano l

Raw Data:



Joshua Thomas
3977033

Experiment 2 Raw data

60% heat

Simple distillation mL	Temp °C
0mL	23.9°C
1	
2	85.9
3	86.4
4	87.0
5	87.5
6	88.1
7	88.9
8	89.3
9	90.6
10	91.9
11	93.5
12	94.4
13	97.4
14	99.8
15	103.3
16	107.7
17	111.7
18	114.8
19	115.8
20	116.3
21	116.7
22	116.8
23	
24	
25	

Observations - seems to be very thick after simple distillation.
- around 28°C temp increase very quickly to 88°C

on is
oilir

polar compounds.

Fractional distillation

ML	Temp °C
0	23.8
1	
2	80.7
3	81.0
4	81.3
5	81.5
6	81.8
7	82.2
8	82.7
9	83.0
10	83.7
11	84.2
12	85.8
13	91.1
14	115.5
15	116.7
16	116.9
17	116.9
18	116.9
19	116.9
20	116.8
21	
22	
23	
24	
25	

Observation; very slowly

recovering

Changed temp from 80 to 85