Experiment 2: Purifying Chemicals by Distillation

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Procedure:

Refer to lab manual. (Dr. Venkateswaran, Rashmi. CHM1321 Organic Chemistry Laboratory. Purifying Chemicals by Distillation. 2014, Exp. 2, p. 19-27. Print.)

Part A: Simple Distillation

- 50 mL of a 50:50 mixture of 2- propanol and 1- butanol was obtained in a graduated cylinder
- The alcohol mixture was poured into a distillation flask and a magnetic stirrer was added
- The distilling apparatus was prepared in the fume hood according to the lab manual and the tutorial from the laboratory website. Extension clamps and clips were used to ensure the stability of the apparatus
- A 50mL graduated cylinder was used as a receiving flask
- A thermometer was obtained and placed through the rubber lid on the distillation head and the tip of the thermometer sat at the junction of the two pipes in the distillation head
- An outlet hose was attached to the top of the condenser, while the other placed in the sink for drainage
- A second outlet hose was attached to the bottom of the condenser while the other end was attached to the water supply
- The heat was turned on to 120 and water was slowly turned on and the distillation begun. The temperature was recorded after every 2mL of distillate.

Part B: Fractional Distillation

- The distillate from Part A was reused as the starting mixture for Part B. Excess 50:50 2-propanol and 1-butanol was added to ensure that 50mL of the solution was put into the distilling flask
- The distilling apparatus was prepared in the fume hood according to the lab manual and the tutorial from the laboratory website. Extension clamps and clips were used to ensure the stability of the apparatus
- A 50mL graduated cylinder was used as a receiving flask
- A thermometer was obtained and placed through the rubber lid on the distillation head and the tip of the thermometer sat at the junction of the two pipes in the distillation head
- An outlet hose was attached to the top of the condenser, while the other placed in the sink for drainage
- A second outlet hose was attached to the bottom of the condenser while the other end was attached to the water supply
- The heat was turned on to 120 and water was slowly turned on and the distillation begun. The temperature was recorded after every 2mL of distillate.

Observations:

Simple Distillation	
Volume (mL)	Temperature (°C)
First Drop	87.3
2	89.7
4	90.7
6	92.0
8	92.5
10	93.4
12	94.4
14	95.4
16	96.4
18	97.3
20	98.5
22	100
24	100.7
26	103.7
28	106.1
30	108.4
32	111.1
34	113.2
36	115.4
38	116.7
40	117.8
42	118.7
44	119.3
46	119.0
48	NA
50	NA

Table 1: Simple Distillation of The 50:50 mixture of 2-propanol & 1- butanol

Fractional Distillation	
Volume (mL)	Temperature(°C)
First Drop	83.3
2	85.6
4	85.9
6	86.9
8	87.9
10	88.3
12	89.4
14	89.7
16	90.8
18	91.9
20	93.2
22	94.6
24	96.8
26	98.7
28	102.0
30	105.6
32	109.7
34	112.8
36	116.3
38	117.5
40	118.4
42	118.8
44	119.3
46	119.3
48	119.1
50	115.0

Table 2: Fractional Distillation of The 50:50 mixture of 2-propanol & 1- butanol

The 50:50 mixture of 2-Propanol and 1-Butanol wad transparent in appearance and had a pungent odor like that of permanent marker.

Magnetic stirrer was mixing the solution

An increase in temperature was observed as time progressed

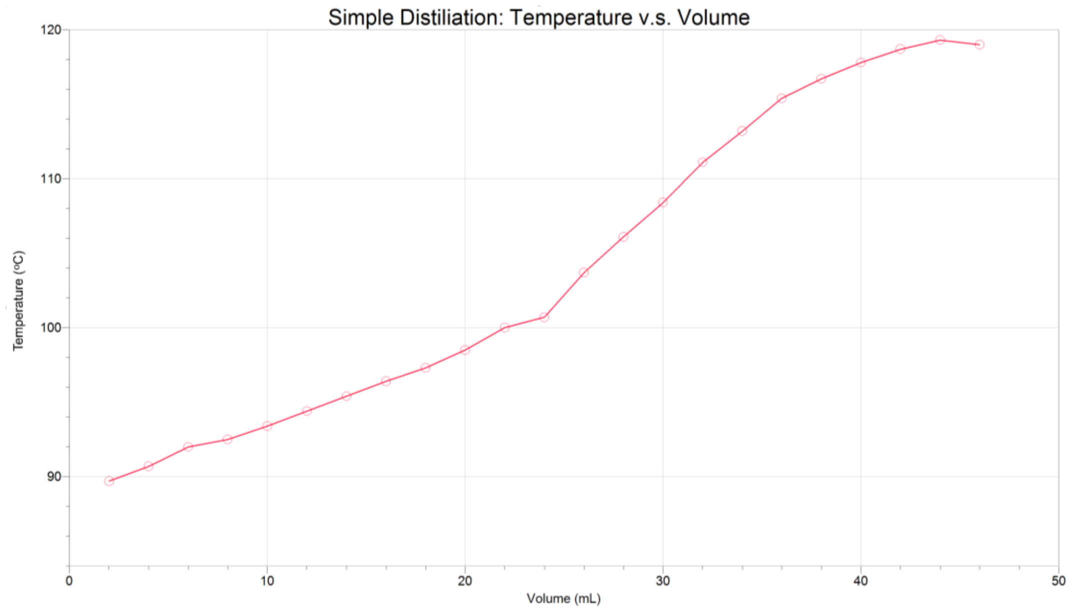

Figure 1: Simple Distillation of The 50:50 mixture of 2-propanol & 1- butanol

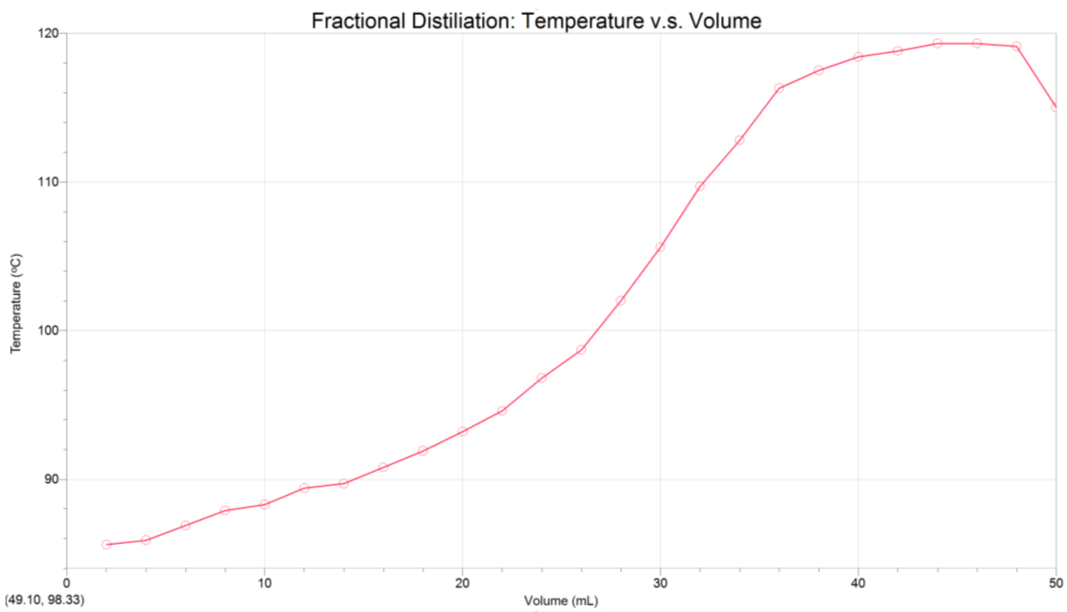

Figure 2: Fractional distillation of The 50:50 mixture of 2-propanol & 1- butanol

While watching the distillation we observed droplets of the solution forming within the fractional column as it was being distilled.

Discussion:

The simple distillation although faster, is not as effecting as the fractional distillation. There is no medium where condensation of the 1-butanol may occur. When looking at both the example in the lab manual and the figure 1 above there is no evident separation of the solution as there is no immediate spike in the graph instead in figure 1 there is a steady slope increase. The rise that does occur in the experimental data occurs at approximately 20 - 25mL, which is to be expected as the original solution was a 50mL 50:50 mixture. When looking at the 2 compounds in order to determine the boiling point you must look at their molecular size as both contain an alcohol functional group. 2-propanol will be the first component to evaporate as its molecules are smaller as they have fewer atoms and thereby will have less intermolecular forces (induced dipoles from Vand der Waal forces) when compared to the 1-butanol, as a result it will take less energy/ heat to change its state from liquid to gas. When looking at the experimental data in figure 1 we observe 2-propanol evaporating at a temperature of approximately 90°C. 1-butanol will have a higher boiling point as it has a greater amount of intermolecular forces, we see its boiling point as being about 120°C.

Part B: Fractional Distillation

With fractional distillations you are able to create a pure mixture by addition of the fractioning column. This addition allows a further separation of the solution by distilling it through a series of cycles of evaporations and condensations occurring due to the packing material. As determined in Part A the 2-propanol will have a lower boiling point and will evaporate fist. The boiling point of 2-Propanol was experimentally determined (figure 2) to be approximately 85°C while the boiling point of 1-butanol was determined to be approximately 120°C. The peak in the temperature that begins at approximately 25mL shows the evident separation between the two components boiling points.

Sources of Error:

When doing this lab there are many areas that may result in slightly skewed results. The first most evident area of error can be seen in the volume of distilled substance. With the simple distillation only 46mL of the initial solution was carried though the entire system. This could be a result of lacking the air tight seal desired between all of the junctions. As a result the vapour was lost into the fume hood. This was corrected for the second experiment. Another source of error that must be accounted for is the human error of measurements. With reading graduated cylinders there are almost always a small amount of error with in the experiment, but this labs measurement errors will be much higher as not only were we're forced to read them at a distance; has to stay out of the fume hood, but also the constant dropping of the distilled solution into the cylinder caused the solution to ripple making it very difficult to read. Next there is the initial amount of heat used to bring the solution to a boil. For Part A of the experiment we started with our power pack turned up to 120. We observed this as going too fast, as one drop per second was the recommended speed in the lab tutorial video. The speed made it harder to read the measure and we promptly turned down the heat to about 110. We believe this is the reason our boiling point for 2-propanol is viewed to be 90°C when in reality it is closer to that of 80°C. This was corrected for Part B where we used a power of 110 initially. With Part B we saw c much closer correlation between the actual boiling point and the boiling point experimentally obtained.

Questions:

1) When distilling you need liquid flowing back through the fractional column in order to get separation of the components. In a fractional distillation the column increases the surface area with the metal packing to obtain a greater number of condensations and vaporizations of the solution. The lower the boiling pointed solution will evaporate up the column while the higher

boiling pointed component will condense and flow back into the flask in a cyclic fashion. This will result in a more effective separation of compounds.

2) It is important to maintain a uniform temperature gradient within a fractioning column because the distillation process relies heavily on the temperature. It is necessary for proper separation of the components. As the vapour moves further up the fractional column the temperature will decrease as it is moving further from the heat source. As a result the component with the higher boiling point will convert back into a liquid and condense at the lower area. This creates a further separation of the two alcohols. This temperature gradient is able to use several condensation and vaporization cycles throughout the column to efficiently separate these components. In the end, there is a maximized amount of the lower boiling point component in the receiving flask.
3) The vapour pressure of benzene at 81°C is equivalent to atmospheric pressure or 101.325kPa. This is because the boiling point of a substance is defined as when its vapour pressure is equal to atmospheric pressure, or the temperature at which the vapour pressure of the liquid equals the pressure applied to the solution.
4) If atmospheric pressure were to increase the substances vapour pressure must increase in order to reach an equivalence point, as a result the boiling point will increase. Appling more pressure to the molecules will give the molecules more rigidity, as a result you will require more energy to break the bonds and turn it to a gaseous state. This can be observed in Gay-Lussas's law because as pressure increases temperature will increase as well.
5) It is important to have cooling water at the bottom of the condenser rather than the top of the condenser because you must ensure the condenser is filled with water and thereby ensuring efficient cooling. When the water is being filled from the bottom position, it is being filled against gravity and fighting against it when being filled. It is important that the condenser is completely filled with water as it maximized the condensation of the vapour back into a liquid form. This will increase the speed and accuracy of the distillation.
6) P mixture = PA* XA + PB* XB

   = (350 mmHg)(0.75) + (150 mmHg)(0.25)

   = 300 mmHg

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

Conclusion:

Simple and fractional distillations were successfully performed on 50:50 mixture of 2-propanol and 1-butanol to separate the two alcohol compounds. The boiling points of the 2-propanol and 1-butanal were determined to be about 85°C and 120°C respectively. The fractional distillation proving to be more accurate.

Raw Data:

...nt 2: Purifying ...micals by Distillation

75% of max power

• connector for thermometer [illegible]

...ml

to droplets upon contact with iron, then drips down

50:50 mixture 2-propanol & 1-butanol

Simple Distillation (initial temp 61.5)

mL	Temperature
2	89.7
4	90.7
6	92.0
8	92.5
10	93.4
12	94.4
14	95.4
16	96.4
18	97.3
20	98.5
22	100
24	100.7
26	103.7
28	106.1
30	108.4
32	111.1
34	113.2
36	115.~~4~~
38	116.7
40	117.8
42	118.7
44	119.3
46mL	119.
48	
50	

Final Volume

Camilo

Fractional Distillation (83.9)

mL	Temperature
2	85.6
4	85.9
6	86.9
8	87.8
10	88.3
12	89.4
14	89.7
16	90.8
18	91.9
20	93.2
22	94.6
24	96.8
26	98.7
28	102.0
30	105.6
32	109.7
34	112.8
36	116.3
38	117.5
40	118.4
42	118.8
44	119.2
46	119.3
48	119.1
50	115

Observations:

→ set to 110 - 120 initial temp 23.6

clear transparent pungent odor like permanent markers