Experiment 2: Purifying Chemicals by Distillation

Procedure

As describe in the Organic Chemistry Laboratory Manual (Durst, T., Scaiano, T., Ogilvie, W., Flynn, A., van Lierop, B., Bush, A., Venkateswaran, R., (2018). Pages 26 to 27).

- For fraction distillation, only 21mL of solution were used because that is all that came out of the simple distillation

Observations

- Simple distillation began faster (drops into gradated cylinder) than the fractional distillation
- Solution dripped faster in the fractional distillation than the simple distillation
- Both 2-propanol and 1-butanol were liquid and transparent substances, with a very strong smell
- As the solution dropped into the graduated cylinder, it appeared to be gel-like as I could see each drop make a ripple and fall to the bottom of the solution that had already accumulate in the graduated cylinder
- During the fractional distillation, the rate at which the drops were falling decreased significantly
- Simple distillation: only 22/225 mL distilled
- Fractional distillation: only 21/22 mL distilled
- As the substance boiled, gas bubbles appeared

Tables

Table 1: Simple Distillation

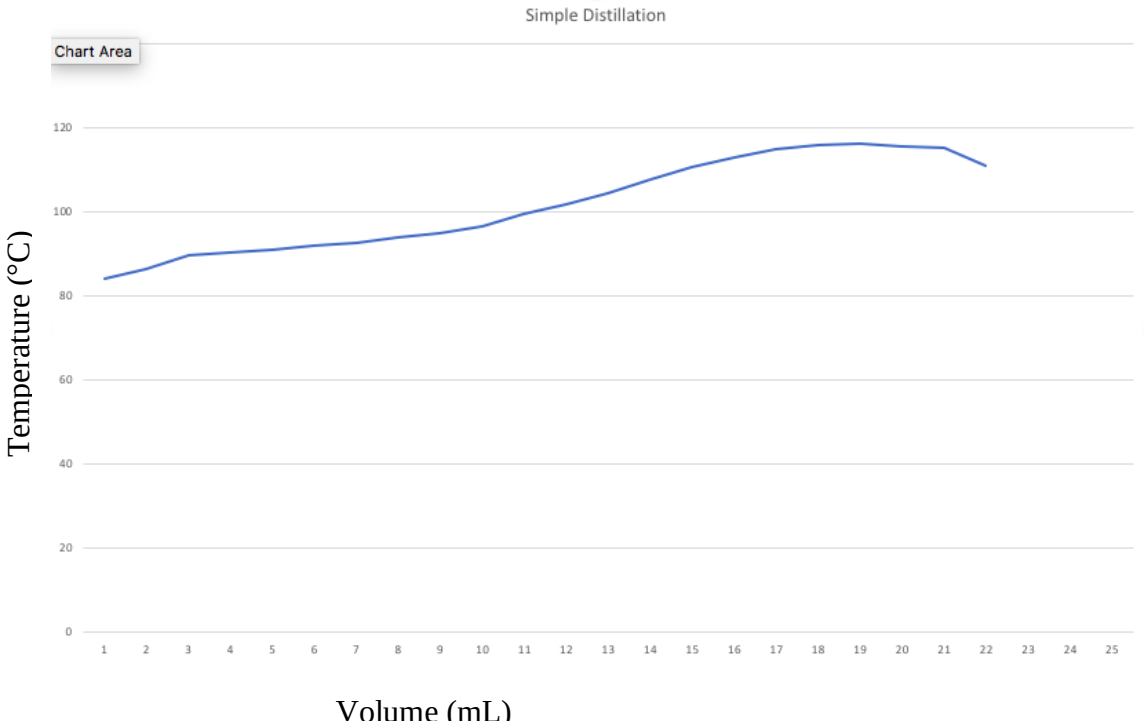
Amount of Substance in Graduated Cylinder (mL)	Temperature (°C)
1	83.9
2	86.4
3	89.6
4	90.3
5	90.9
6	91.8
7	92.5
8	93.8
9	94.9
10	96.6
11	99.6
12	101.6
13	104.5
14	107.7
15	110.6
16	113.0
17	114.7
18	115.7
19	116.0
20	115.5
21	115.0
22	110.8
23	-
24	-
25	-

Table 2: Fractional Distillation

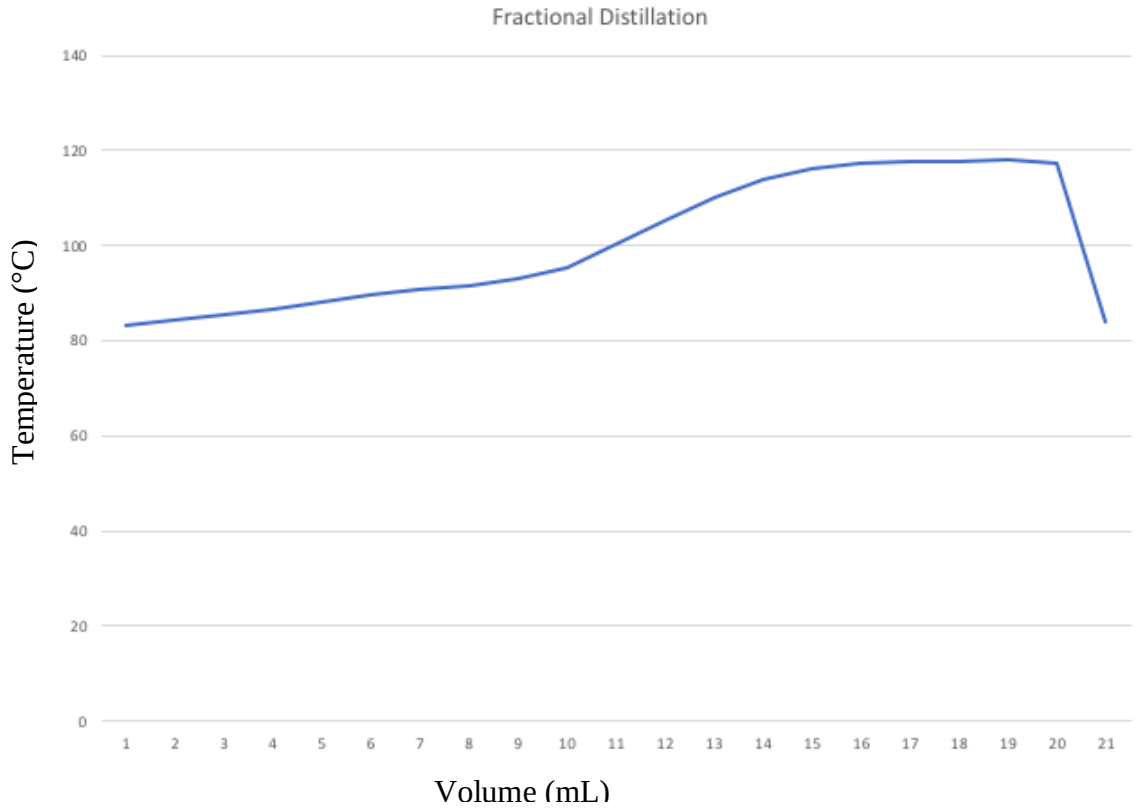
Amount of Substance in Graduated Cylinder (mL)	Temperature (°C)
1	83.2
2	84.3
3	85.6
4	86.8
5	88.0
6	89.7
7	90.7
8	91.6
9	92.9
10	95.5
11	100.3
12	105.1
13	110.3
14	113.9
15	116.0
16	117.2
17	117.6
18	117.8
19	117.9
20	117.4
21	84.1
22	-
23	-
24	-
25	-

Graphs

Graph 1: Simple Distillation



Graph 2: Fractional Distillation



Discussion

- Distillation is a method of separating two or more compounds based on their differences in boiling point
- It is used as a means of purification, in which liquids are vapourized and the vapours are condensed and collected
- There are two common methods of distillation, simple distillation and fractional distillation
- Although they are similar, simple distillation requires an easier apparatus setup and tends to be a quicker process than the fractional
- The equipment used is very similar, except the fractional distillation requires a fractional column between the distillation head and the distilling flask
- Fractional distillation gives a better separation of the compounds in the mixture, by allowing the undesirable compound to condense and only the more volatile compound to go out
- The choice of whether to use simple or fractional distillation depends on the liquid involved, and the difference in boiling point of the compounds in the mixture
- Simple distillation is better suited for when the two compounds have a large difference in boiling point (>100 ℃)
- Fractional distillation is better suited when the two compounds have a smaller difference in boiling point (<100 ℃), it gives a much better/more clear separation between the two compounds and can more easily purify complex mixtures than simple distillation
- In this experiment, the two substances used were 2-propanol and 1-butanol
- 2-propanol has a boiling point of 83℃ and 1-butanol has a boiling point of 118 ℃, making the difference in boiling point 35℃, therefore fractional distillation is the more accurate/appropriate distillation method for this mixture
- in the simple distillation graph, the temperature remains relatively stable around 90℃ until about 9mL is distilled then it begins to increase more significantly, until it plateaus at 17mL around 115℃, and then begins to drop at 22mL with a temperature of 110.8℃
- only 22mL/25mL of the substance distilled
- in the fractional distillation graph, a slow and study increase in temperature occurs from 83℃ to 92℃, then at 10mL the temperature begins to increase more significantly until it plateaus at around 117℃ at 16mL, then drops quite drastically to 84.1℃ at 21℃
- only 21mL/22mL of the substance distilled
- As the distillation proceeds and the lower boiling components are removed, more theoretical plates are needed to reach the desired purity
- The fractional column has a certain number of theoretical plates that it can handle, so at a certain point it can no longer handle the separation. This results in the mixture being distilled until the entire lower boiling component has been removed. At that point the pure second component begins distilling as evidence by the second temperature
- Based off the data collected the ratio of pure components is 11mL of 2-propanol : 14mL of 1-butanol
- The graphs show that the fractional distillation line has a steeper slope than the simple distillation line. This proves that fractional distillation is the more appropriate method of separation for these compounds, because there is a distinct separation between the 1-propanol and the 2-butanol, represented by the steep slope

- The shallow slope of the simple distillation shows an unclear/poor separation because there is not distinct cut off between the 1-propanol and the 2-butanol
- The boiling point of each substance is represented by the plateaus on the graphs
- The graph of the simple distillation shows a plateau around 90°C, representing the boiling point of 2-propanol, although, it is not accurate as the actual boiling point is 83°C; another plateau is seen at 115°C, representing the boiling point of 2-butanol, but again is not exactly accurate as the actual boiling point is 118°C.
- The graph of the fractional distillation shows more accurate boiling points for 1-propanol (83-85°C) and 2-butanol (117°C), as well as the distinct separation by a steep slope
- The addition of metal sponge and insulation from tinfoil on the fractional column increases the surface area in the fractional distillation causing a better separation
- Sources of error may include: incorrect measurement of the substance, which could have been why less than 25mL distilled; human error in watching the graduated cylinder fill, noting inexact measurements; improper insulation of the fractional column with tinfoil

Questions

1. Having the liquid flow back through the fractional column is necessary for the vapour to condense. Otherwise the temperature would not be cold enough and condensation would not occur. The vapour becomes more enrich with the lower boiling point compound and it will travel up until it reaches the height where the temperature is low enough that the vapour can condense and turn into a liquid. The purpose of flowing the liquid back into the column is to get a more accurate separation of the solution being distilled by the temperature gradient in the column allowing the molecules with a higher boiling point to re-condense. It then drips down while the lower boiling point molecules are still able to rise up to be distilled by the warm vapour rising up. Once the temperature reaches the boiling point of the higher boiling point molecules, they will continue to rise but all the lower boiling point molecules will already be fully distilled. That is why flowing the liquid back into the fractional column is important.

2. It is vitally important to maintain a uniform temperature gradient in the fractional column so that the rising vapour will not condense before it reaches the condenser. If the column varies in temperatures throughout, the vapour would condense and the liquid will drip back down, not allowing for proper separation of the mixture. This assisted by the column being isothermal, as a particular fraction can travel up the column at a particular temperature, without condensing and dropping back down.

3. The vapour pressure of benzene at 81 degrees Celsius is 1 atm. The boiling point of a substance is when the vapour pressure is equal to the pressure surrounding the liquid.

4. Increasing atmospheric pressure will cause the boiling point of the liquid to increase as well because the boiling point is dependent on the pressure.

5. The purpose of the condenser is to cool the vapour so that it turns into a liquid. Therefore, the cooling of the water must enter from the bottom of the condenser so that there is cool

water always flowing around it. If the water were to enter from the top it wouldn't fill the condenser and the condenser couldn't do its job.

6. Using Raoult's law:

$P_{total} = (P_A \times N_A) + (P_B \times N_B)$
$= (350 \text{ mm Hg}) \times (3/4) + (150 \text{ mm Hg}) \times (1/4)$
$= (262.5) + (37.5)$
$= 300 \text{ mm Hg}$

Therefor the vapour pressure of 3:1 of A and B at 95°C is 300 mm Hg.

Citations

Organic Chemistry Laboratory Manual (Durst, T., Scaiano, T., Ogilvie, W., Flynn, A., van Lierop, B., Bush, A., Venkateswaran, R., (2018). Pages 13 to 19).