Lab Report #2

Purifying Chemicals by Distillation

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Introduction

In this experiment we use two different methods of distillation that are commonly used to separate two compounds in a mixture: simple distillation and fractional distillation.

Using a simple distillation the mixture is placed in a distillation flask and is then heated up. What happens first is that the compound in the mixture with the lowest boiling point will change its phase from liquid to vapor. The vapor produced migrates up and enters the condenser tube, and with the help of the cold water flowing will change its phase back to liquid phase and will slowly drip into the receiving flask. This way, we are separating the two compounds based on their different boiling points.

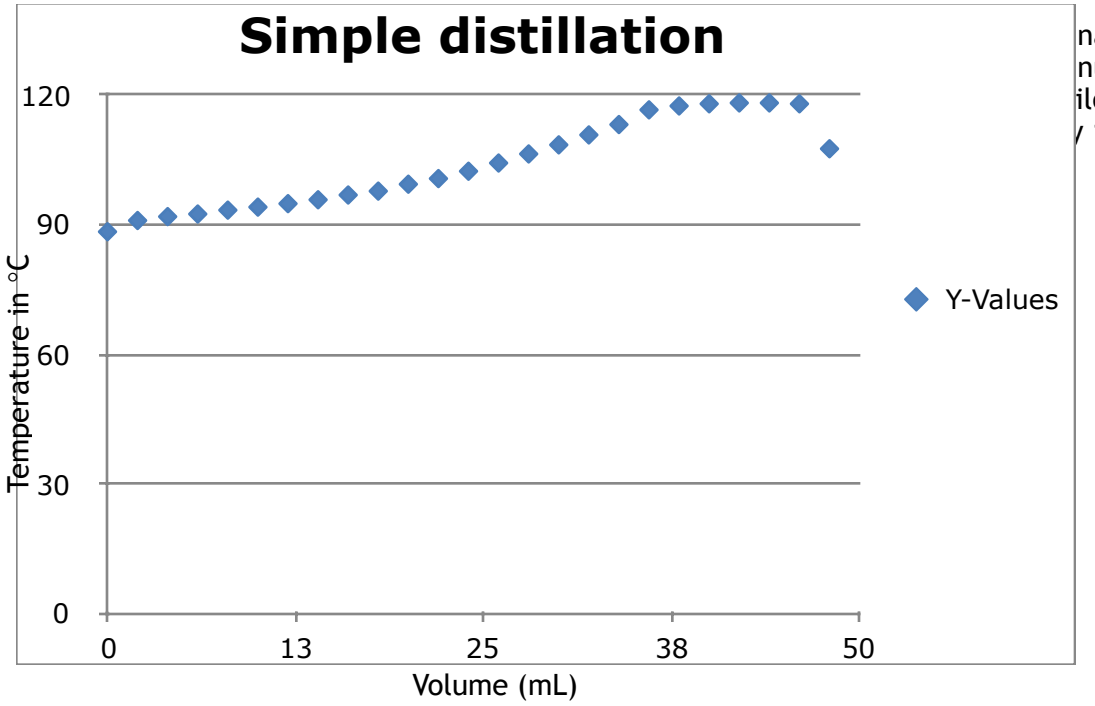
Fractional distillation is exactly the same concept as the simple distillation. However the difference is that the gas does not migrate up a glass normal glass column into the condenser but through a fractionating column that is added. The purpose of the fractioning column is to provide a surface for the vapor to condense: in fact, the surface of the area is thanks to packing, which is a material with a high surface area in the fractioning column.

Procedure

Part A: Simple Distillation

- We used the lab manual's procedure on how to set up the apparatus as presented on page 24, assuring that the apparatus is tightly secured with clamps, and having put grease around the openings of every tube as well as the distilling flask.
- We used a 100mL distilling flask and the receiving flask used was a 50mL graduated cylinder.
- The distilling flask contained 50mL of a 50:50 mixture of propan-2-ol and 1-butanol and a magnetic stirrer to generate a vortex to keep the mixture mixed. The magnetic stirrer was set to 7.
- The condenser had two rubber tubes, one entering the water into the condenser from the bottom and the other allowing the water to leave through the top.
- The heating mantle was initially set to 80.
- To help speed up the process, we were advised to wrap aluminum foil around our apparatus in order to speed up the heating.
- When everything was ready, we waited and took the temperature at the first drop; and so on we recorded after each 2 mL portion of distillate is collected, until the distillation was complete.

Part B: Fractional Distillation



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- The procedure is essentially the same as for the simple distillation, however here we add a fractioning column packed with the “packing” material, instead of using a standard glass tube when assembling the apparatus.
- The heating mantle in this case was initially set to 85. However, we raised the energy to 95 at 24 mL because our distillation was taking too long, even with the aluminum wrapped around it.
- Once again, we took the temperature for every 2mL recruited after the initial drop and until the distillation was complete.

Observations

Simple distillation

Volume (mL)	Temperature (°C)	Volume (mL)	Temperature (°C)
Initial drop	88.4	26	104.3
2	91.0	28	106.4
4	91.9	30	108.5
6	92.5	32	110.8
8	93.4	34	113.2
10	94.1	36	116.6
12	94.9	38	117.5
14	95.8	40	118.0
16	96.9	42	118.2

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Initial drop	81.1	26	103.3
2	83.9	28	114.9
4	84.4	30	117.1
6	84.8	32	117.9
8	85.2	34	118.2
10	85.6	36	118.3
12	86.1	38	118.4
14	86.8	40	118.4
16	87.6	42	118.5
18	88.8	44	118.5
20	90.0	46	118.4
22	91.9	48	---
24	95.2 *** Here we raised the energy from 85 to 95	50	---

Graph obtained for the fractional distillation

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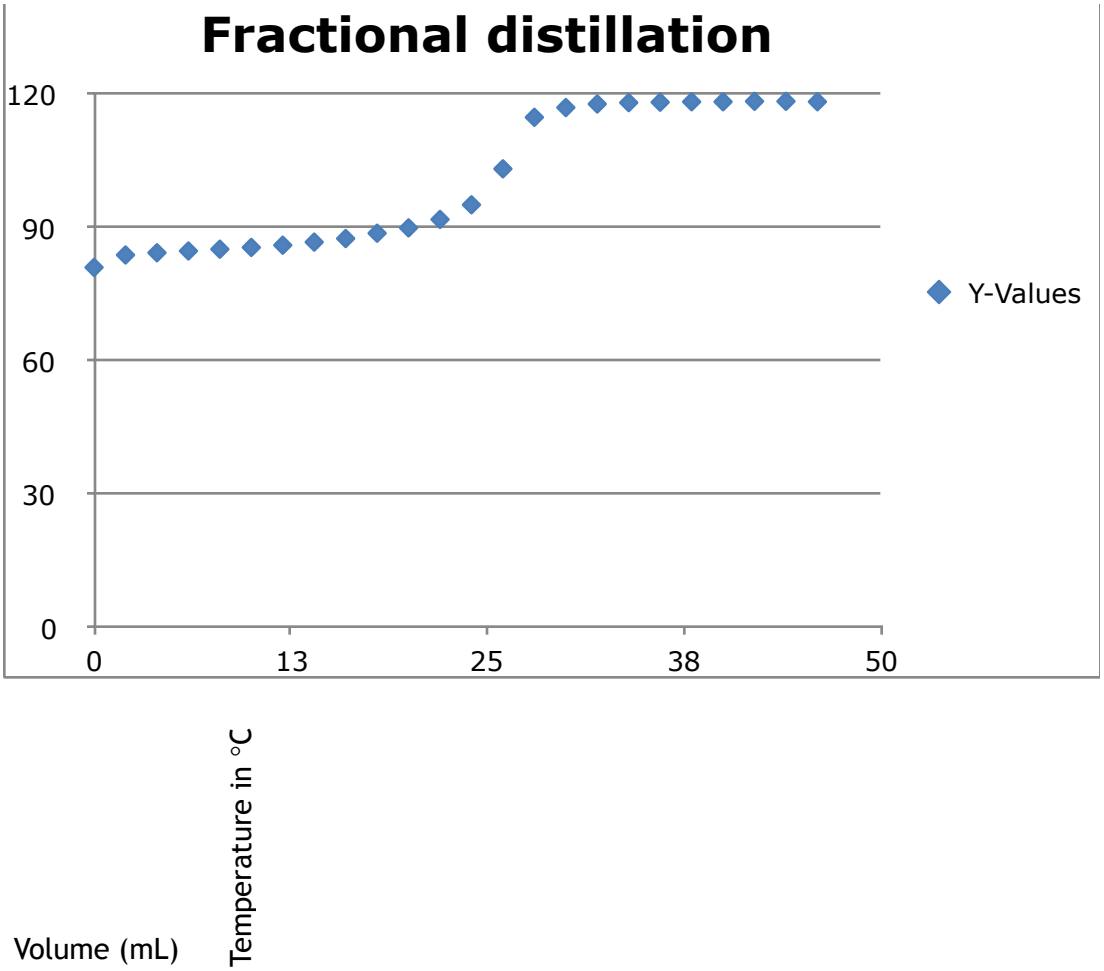

Note: For the purposes of the graph, the initial drop is considered to be at 0mL.

Discussion:

Simple distillation

For the simple distillation, analyzing the data we have obtained, we can see that the slope for the simple distillation is almost linear. In fact, the temperature rises slowly and steadily according to the amount of distillate obtained. The slope goes up gradually as the distillation proceeds and is not very curved. This corresponds to what a simple distillation is supposed to show us in general: as the mixture is being heated, the temperature is not constant but increases through the whole process, because the composition of the vapor varies constantly. Both components are being sent into the receiving flask.

In fact, Raoult's law states that the total pressure exerted by the mixture is equal to the sum of the partial pressures of the components (the partial pressure of each component being the partial pressure of the component in the mixture of vapors multiplied by the mole fraction of the compound in the mixture). The law suggests that the vapor contains more of

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Propan-2-ol than there is in the liquid, since it is more volatile (because it has a lower boiling point of 85.2 degrees). However, there is still a gradually increasing percentage of butan-1-ol composing the vapor as the temperature goes up and most of the propan-2-ol is being retrieved in the receiving flask (as we can in Figure 4 on page 21 of the lab manual). This slow alteration of the composition of the vapor directly reflects the composition of the mixture in the receiving flask. This signifies that the receiving flask that we are obtaining is not completely pure, since there is a small portion of butan-1-ol that also entered the receiving flask.

The process of simple distillation is faster. However, this is not necessarily a good thing, as we could not separate the two liquids during the distillation; there was never only propan-2-ol or 1-butanol in the vapor for the simple distillation. So, although there is mostly propan-2-ol being collected at the beginning of the distillation, and mostly 1-butanol in the end, the separation will still not be efficient because the composition of the vapor varies continuously.

Fractional Distillation

For the fractional distillation, the slope rises very slowly up until around 91 degrees (25 mL) where there is a drastic increase in the temperature (91 degrees is a little bit higher than propan-2-ol's boiling point, which is around 82 C). I should note that we raised the energy (from 85 to 95) of our mantle at around 91 degrees because our heating mantle wasn't functioning properly. Also, the increase of the temperature between the initial drop and our 2mL is probably due to the residue from the simple distillation. Normally, the temperature should have been more constant around the boiling point of propan-2-ol (almost like a straight line), and only once it has been removed from the solution should the temperature have started increasing to butan-1-ol's boiling point temperature.

In the fractional distillation, we can clearly see the separation in the graph. In fact, the temperature raises slowly up until 91 degrees, and once all of the propan-2-ol has been collected, the temperature quickly rises to the boiling temperature of the second component, 1-butanol, which is around 118 degrees, and from then on the temperature was stable and stopped increasing. In this method, the separation is more efficient: we know almost precisely when propan-2-ol has fully been collected, and when the collection of 1-butanol begins, and the composition of the vapor is not constantly changing like for the simple distillation. At the beginning the vapor is only composed of propan-2-ol, and after it has been collected, butan-1-ol begins to be collected.

Comparing these two methods, fractional distillation is clearly the most efficient, even if it takes more time than the simple distillation. Fractional distillation is more efficient because it is as if there are multiple simple distillations taking place at once. Simple distillation also can be efficient if it is repeated numerous times, however the advantage of fractional distillation is that one distillation is enough to have a good separation of the components in the mixture.

Simple distillation is a less good choice in this case, especially since the boiling temperatures of propan-2-ol and 1-butanol are only about 35 degrees apart, and it is better

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to have a large difference in boiling point between two liquids to have a more successful separation (over 100 degrees in difference is preferred as stated in the lab manual). Simple distillation also is known to work better when trying to separate a liquid and a solid, or also in a solution where one of the two components is only presents in traces.

Questions:

1) The liquid that collects on the packing within the fractioning column must flow back through the fractioning column as the remaining vapor continues to rise in order to get the separation of the components during a fractional distillation. It is the liquid that has the lowest boiling point that will continue through the apparatus into the condenser and the liquid with the higher boiling point will return into the distillation flask. The heat from the rising vapor coming from below is responsible for the re-vaporization of some of the condensation on the packing material, mostly of the liquid with the lower boiling point. The liquid that condensates on the packing material and drips back down is the liquid with the high boiling point. This way, after multiple cycles, the receiving flask will have more of the liquid with the lower boiling point and the initial flask will be enriched in the liquid with the higher boiling point, allowing an efficient separation of the liquids of our initial mixture.

2) A smooth and uniform temperature gradient is important in a fractional column variations in temperature in the fractioning column can alter the distillation process. For example, if the temperature is too high, the liquid that is supposed to be flowing back into the distillation flask will be prevented from doing so. The liquid with the higher boiling point can reach the condenser, which can ruin the purity we are trying to obtain. The insulation allows the temperature to be stable in the fractioning column and helps our separation be more efficient.

3) Normally, if a liquid is boiling, that means that it has reached the pressure of its environment. Considering this, we can deduce that the vapor pressure of benzene at its boiling point (81°C) is 1atm.

4) An increase in atmospheric pressure results in an increase in temperature, as stated by the ideal gas law:

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5) It is important to have cooling water enter the bottom of the condenser and not the top because otherwise the condenser will not be completely full of water. In fact, if the water came in from the top, it would quickly slide down the condenser and exit from the bottom hole. If the water enters from down, the water is forced to stay and fill up the condenser throughout the whole distillation.

6) Raoult's law states that the total pressure (P_{total}) exerted by the mixture is equal to the sum of the partial pressures of the components.

$P_A = (P_{A^*})^*(N_{A^*})$

$P_B = (P_{B^*})^*(N_{B^*})$

We have a 3 :1 mixture of A and B at 95 C. Considering that $N_A + N_B = 1$, the mole fraction of A is $N_A = 0.75$, while the mole fraction of compound B is $N_B = 0.25$.

$P_{TOTAL} = (P_{A^*})^*(N_{A^*}) + (P_{B^*})^*(N_{B^*})$s

$= (350^*0.75) + (150^*0.25)$

$= 300$ mm Hg

The vapor pressure of a 3:1 mixture of A and B at 95 C is 300 mm Hg