Experiment #2

Purifying Chemicals by Distillation

CHM1321

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Procedure:

Simple Distillation

- A simple distillation apparatus was assembled in the fume hood according to the diagram in the lab manual.
- Approximately 50mL of a 50:50 mixture of 2-propanol and 1-butanol was added to the distillation flask.
- The distillation head, condenser and takeoff adapter were all added to the set up. A stand and stir plate were used to hold the receiving 50mL graduated cylinder and the 100mL distillation flask. All clamps were used to properly hold the apparatus at appropriate spots such as holding the distillation flask.
- The temperature probe was inserted down the distillation head resting just in the middle the distillation head.
- Tubes were connected from one of the water sources to the condenser, and also from the apparatus to the sink.
- Water flow was turned on and the solution were slowly distilled using low heat of about 50-60 degrees and stirred using a magnetic stirrer.
- Aluminum foil was wrapped around the distillation flask to avoid any heat loss.
- The temperature was recorded using the thermometer for every 2mL of distillate collected in the distilling graduated cylinder.

Fractional Distillation

- The still was disassembled, but the stirrer and mantle were kept in place.
- The distillate in the graduated cylinder was disposed of and another 50mL of a 50:50 mixture of 2-propanol and 1-butanol was added to the distillation flask.
- A fractionating column was insulated with aluminum foil then assembled onto the apparatus.
- The fractionating column and the flask and other parts of the apparatus were clamped down.
- The temperature probe was inserted down the distillation head resting just in the middle the distillation head.
- Tubes were connected from one of the water sources to the condenser, and also from the apparatus to the sink.
- Water flow was turned on and the solution were slowly distilled using low heat of about 50-60 degrees and stirred using a magnetic stirrer.
- Aluminum foil was wrapped around the distillation flask to avoid any heat loss.
- The temperature was recorded using the thermometer for every 2mL of distillate collected in the distilling graduated cylinder.
- All parts were cleaned, and the distillate and mixture disposed of in the waste container.

General Observations:

- The mass of the graduated cylinder before adding the distillate was 73.95g.
- The mass of the graduated cylinder after adding the distillate was 103.12g.
- The 50:50 mixture of 2-propanol and 1-butanol had an alcoholic smell, was clear, non-viscous, colourless and a liquid.
- The initial temperature when the thermometer was first inserted was about 20 degrees celsius.
- The distillate pure substance was a liquid that was clear, colourless, and had a oily viscosity.

Table 1: Simple Distillation

Volume of Distillate (mL)	Temperature (°C)
2	53.0
4	56.2
6	56.8
8	55.1
10	59.6
12	60.1
14	61.5
16	62.5
18	62.9
20	62.9
22	62.4
24	63.5
26	64.3
28	65.1
30	66.0
32	66.2
34	66.6

36	72.6
38	74.2
40	72.9

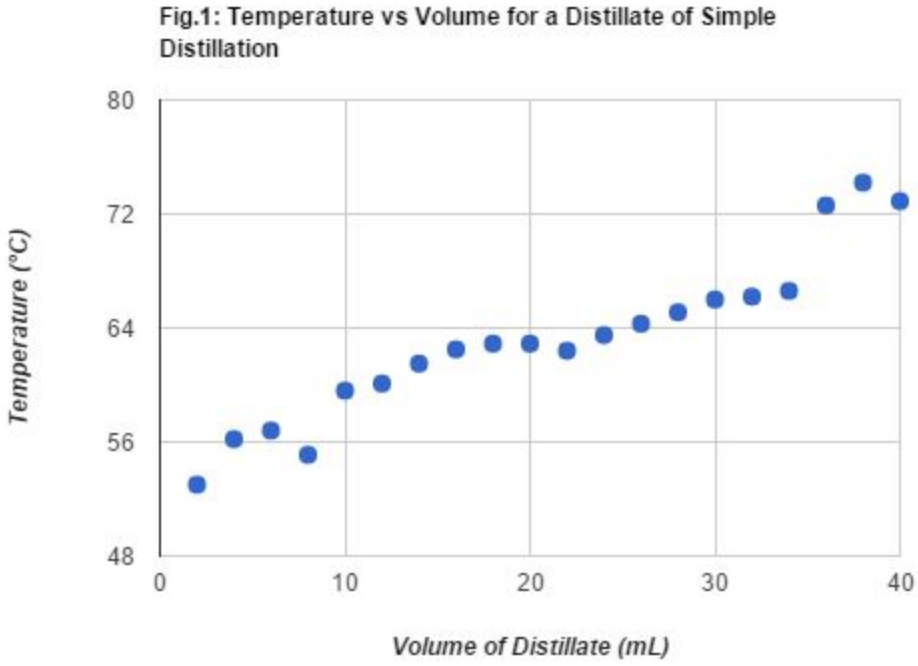

Fig. 1: This graph depicts the relationship between the temperature and volume of a distillate for Simple Distillation. The temperature increases with a very flat slope, while at the two extremes at 10mL and 36mL the temperature increases sharply.

Table 2: Fractional Distillation

Volume of Distillate (mL)	**Temperature (°C)**
2	70.6
4	80.1
6	80.8

8	81.4
10	81.9
12	81.7
14	81.8
16	82.2
18	82.6
20	83.1
22	83.8
24	83.7
26	76.0
28	68.0
30	62.6
32	65.4
34	108.2
36	114.8
38	115.9
40	116.1
42	115.9
44	107.6
46	97.6

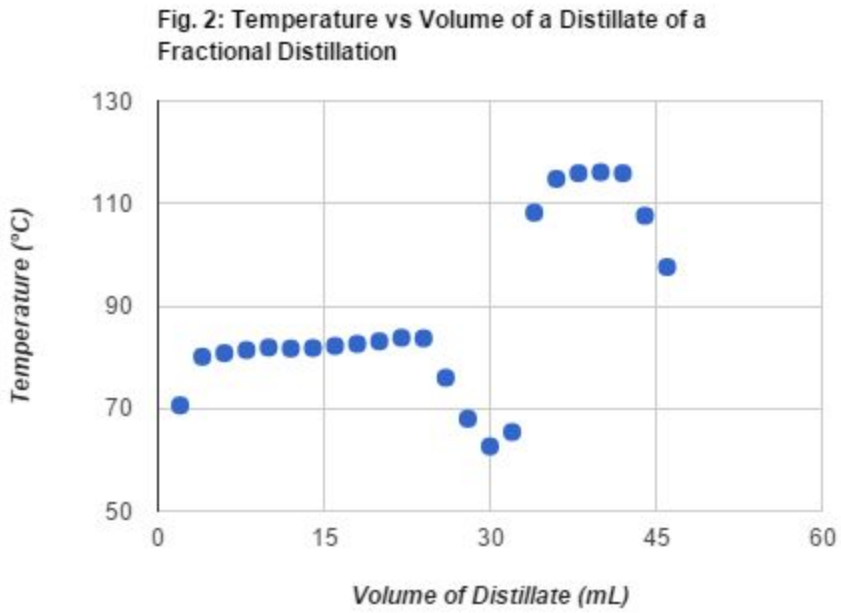

Fig. 2: This graph depicts the relationship between the temperature and volume of a distillate. The temperature is held constant until 24 mL where the temperature starts decreasing. At 34 mL the temperature increases sharply, indicating the distillation of substance B.

Discussion:

The shape of each temperature curve that was prepared, measuring the temperature vs volume of the distillate obtained via both simple distillation and fractional distillation, matched the theoretical results that were thought to be obtained.

Firstly, we take a look at simple distillation. In simple distillation, the vapourized liquid is carried upwards through the distillation head into the condenser. Once there it cools on contact with the condenser, which is pumped full of water, and returns back into liquid form, running down the condenser as drops, and is collected in the receiving flask, or in our case, the graduated cylinder. The distillate collected in the graduated cylinder is the pure substance obtained from the mixture put into the distillation flask; let us call this substance A. We call this substance A because at the beginning of the distillation, the substance with the lowest boiling point will be removed first and enriched in the receiving flask. In our case Substance A was the 2-propanol, while substance B, the component of the mixture still remaining in the distillation flask was the 1-butanol with a higher boiling point.

On the hand we have fractional distillation. This form of distillation follows the exact same set-up as the simple distillation however, it differs through the use of a fractionating column. This column is induced to improve separations between the two components of the mixture at use. This is achieved primarily by increasing the height of the apparatus, in turn

creating a greater temperature gradient. This gradient can be kept constant by applying a thin layer of aluminum foil or any other insulating material to keep the column from losing heat. The column is also packed with an inert material to allow for the vapour to condense onto it when the distillation is occurring. Moreover, the inert material also acts to increase the surface area to which the vapour can condense on. As the vapour rises in the fractionating column, it comes in contact column and condenses on the packing. Heat from the rising vapour below causes some of this condensate to vapourize again; this allows for more of Substance A, the component with the lower boiling point, to be vapourized. Because of this, substance B, the component with the higher boiling point will condense and drip back down while substance A will move towards the condenser and become enriched in the receiving container. This form of distillation allows for greater separation of mixtures and therefore resulting in purer products.

In comparing the two forms of distillation, one can conclude that both methods have their advantages and disadvantages. Simple distillation is occurs much faster and can separate the two components much more quickly. Fractional distillation occurs much slower, however, in terms of efficacy, fractional distillation allows for a greater separation of both species. This advantage was described above, and is attributed to the fractionating column which creates a temperature gradient in order to better separate the products. On the other hand when using simple distillation, the distillate will contain a greater mixture of both substance A and B.

The graph of Fig. 1 shows a linear increase in temperature as volume of distillate increases. At both extremes of the graph we can see that the temperature stabilizes. This occurs because the temperature of a pure substance should be constant. In this case, when the distillation has finished with substance A, the temperature levels off. Our graph does not exactly match the ideal curve but if outliers are ignored it is almost accurate.

The graph of Fig. 2 shows a near constant temperature up to 34 mL where the temperature suddenly increases drastically. At this point we know that the distillation of the first component is finished and the distillation of substance B is beginning.

This lab was very tedious and required a lot of attention to detail especially when setting up the apparatus. Some sources of error included loss of heat through the distillation flask, escaping vapour through the apparatus especially in the distillation head. To improve this lab, aluminum foil acted as an insulator and was used to cover the distillation flask and the fractionating column. Moreover, the fume hood was closed in order to prevent vapour loss and heat loss as well.

When looking at the distilling method which was most effective in purifying chemicals, the fractional distillation was advantageous.

Questions:

1. In order for fractional distillation to be a method of distillation which is high in efficacy, the liquid must flow back through the fractionating column to get separation of the components during the distillation. This is is required because as the component with the lower boiling point moves upward towards the fractionating column, the component with the higher boiling point will condense and drop back into the the distillation flask as a liquid, it will come in contact with the fractionating column, re-vaporize and become enriched in the graduated cylinder. If the liquid did not flow back through the column there would be no separation of the mixture. This can also lead to flooding which would disallow for distillation to occur.

2. The fractionating column has a temperature gradient which is very important to allow the vapour to rise and re-vaporize and for the liquid to flow back down in which allows separation of the components. The fractionating column increases the height of the apparatus and therefore distancing the vapour from the heat source, resulting in an overall decrease in temperature. As the vapour containing both components of the mixture rises, it condenses on the packing material on the fractionating column. As the component with the lower boiling point condenses, the one with the higher boiling point will drip back down as a liquid in the distillation flask. Because of the temperature gradient, the condensation that occurs in the fractionating column will occur at different heights. After the minicycle of condensation occurs multiple times, the highest point of the fractionating column, that is the point with the lowest temperature, will have a high concentration of component A condensing, and in turn more of substance A being collected in the receiving flask. This allows for a very effective separation of the two components.
3. Since the boiling point of benzene is 81 degrees celsius, this means that the vapour pressure of benzene is equivalent to the atmospheric pressure. The atmospheric pressure is equal to 1.013×10^5 Pa and therefore the vapour pressure is equivalent.
4. In question 3, we know that the vapour pressure is equal to the atmospheric pressure. Therefore, if the atmospheric pressure were to increase, the vapour pressure would increase as well. The boiling point of a gas is when, the atmospheric and vapour pressure are equal. From this we can deduce that the temperature at which the boiling point occurs will increase as well. This is proven through the ideal gas law, PV=nRT.

$$P_1/T_1=P_2/T_2$$

From this equation we can see that if pressure increases, the temperature must increase as well.

5. It is important to have water entering from the bottom so that the lower region of the condenser is filled with cooled water, in order for the vapour to condense and be collected in the receiving graduated cylinder. If the water were entering from the top, the condensed vapour would drip back it the distillation flask.

6. Raoult's Law states that:

$P_{mixture} = P_A \circ X_A + P_B \circ X_B$
$= (350mmHg)(.75) + (150mmHg)(.25)$
$= 300mmHG$

The vapour pressure of the 3:1 mixture of A and B at 95 degrees celsius is 300mmHg.