

Lab #2
Purifying Chemicals by Distillation

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Presented to: Joshua Derasp
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Introduction

- Distillation is a process often used to separate two compounds based on their boiling points, or in other words purify a single compound.
- A 50:50 mixture of 2-propanol and 1-butanol was separated based on the boiling points of the 2 different compounds by using a distillation procedure
- The distillations were conducted both with and without a fractional column, i.e. a simple distillation as well as a fractional distillation.
- The addition of the fractional column to the apparatus better separated the compounds by creating mini distillations within the column to result in a purer end product.
- The apparatus was constructed to the needs of the distillation in order to properly complete the experiment.
- The solution was added to the flask, heated, vaporized, condensed, and collected while the boiling temperature was observed.

Procedure & Observations

Fractional Distillation:

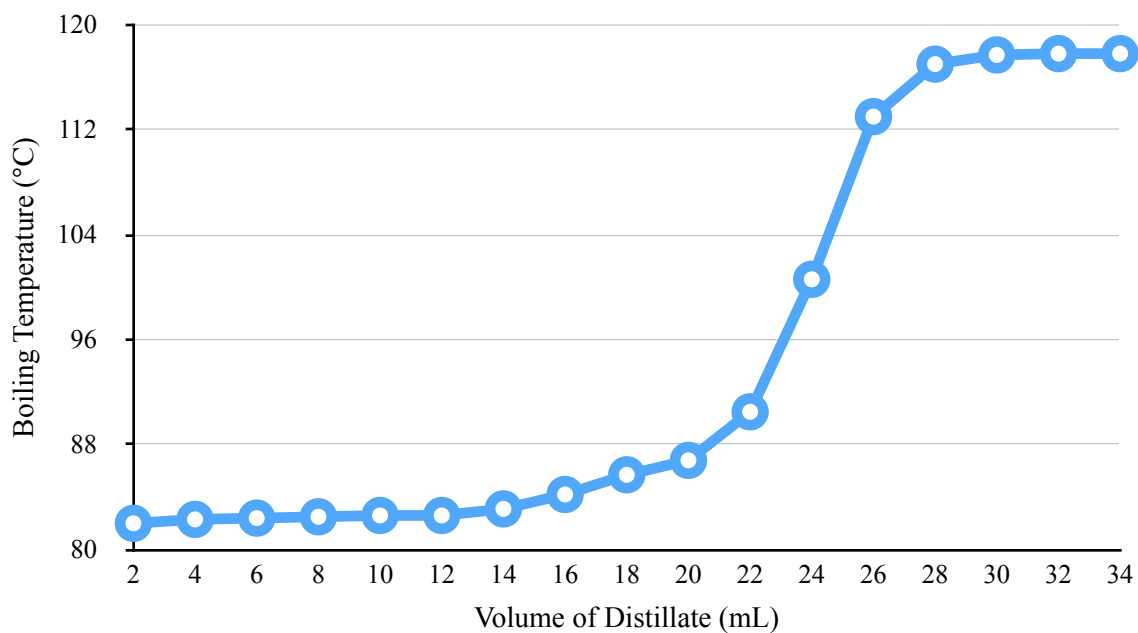
1. 50mL of 50:50 mixture of 2-propanol and 1-butanol was added to a distillation flask.
2. A magnetic stirrer was acquired from the TA and placed in the distillation flask along with the 50:50 solution.
3. The apparatus was constructed as described in the Lab Manual, including a fractional column.
 - Aluminium foil was wrapped around the apparatus from the extension clamp on the distilling flask to the thermometer adapter to ensure no vapour was leaked from the apparatus.
4. A 100mL graduated cylinder was placed under the vacuum take-off adapter and was used as the receiving flask.
5. The hot plate was turned on and set to a medium-low heat (about 4).
 - The as the solution was heated it began to boil, the magnetic stirrer stirred the solution at a constant pace.
6. As the distillation progressed, the temperature indicated on the thermometer was recorded after every 2mL of the distillate was collected.
 - The distillation took much longer to begin to produce distillate than the simple distillation
 - The distillate was collected at an extremely slow pace, about 1 drop every 5 seconds
 - Because of the extremely slow distillation, data for the fractional distillation was collected from Peter Smith (6768456) and Jennifer Newell (7265443).

Table 1; Boiling temperature (°C) vs volume of distillate (mL) for the Fractional Distillation

Volume (mL)	Temp. (°C)	Volume (mL)	Temp. (°C)
2	82.0	20	86.8
4	82.3	22	90.5
6	82.4	24	100.6
8	82.5	26	113.0
10	82.6	28	117.0
12	82.6	30	117.7
14	83.1	32	117.8
16	84.2	34	117.8
18	85.7		

7. After the distillation was completed, the distilled material was returned to the proper waste bottle and the heat shut off.
8. The apparatus was carefully taken apart and all pieces were returned to the organic chemistry locker.

Figure 1; Boiling temperature (°C) vs volume of distillate (mL) for the Fractional Distillation



Simple Distillation:

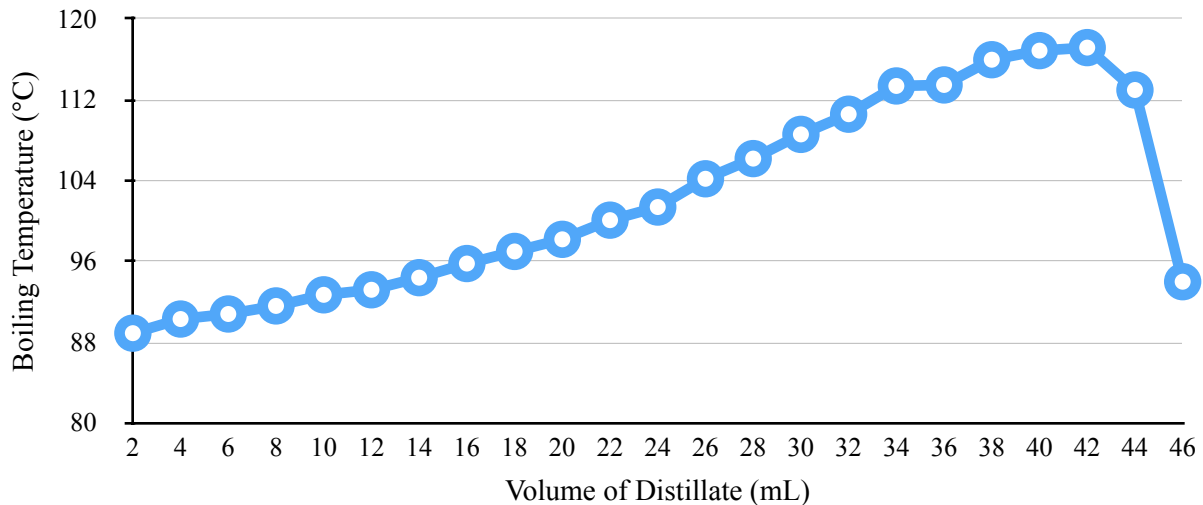
1. 50mL of 50:50 mixture of 2-propanol and 1-butanol was added to a distillation flask.
2. A magnetic stirrer was acquired from the TA and placed in the distillation flask along with the 50:50 solution.
3. The apparatus was constructed as described in the Lab Manual.
 - Aluminium foil was wrapped around the apparatus from the extension clamp on the distilling flask to the thermometer adapter to ensure no vapour was leaked from the apparatus.
4. A 100mL graduated cylinder was placed under the vacuum take-off adapter and was used as the receiving flask.
5. The hot plate was turned on and set to a medium-low heat (about 4).
6. As the distillation progressed, the temperature indicated on the thermometer was recorded after every 2mL of the distillate was collected.
7. Data was collected from Shawna Rieux (7706547) and Katherine Jennings (7824410)

Table 2; Boiling temperature (°C) vs volume of distillate (mL) for the Simple Distillation

Volume (mL)	Temp. (°C)	Volume (mL)	Temp. (°C)
2	88.9	26	104.2
4	90.3	28	106.2
6	90.8	30	108.6
8	91.6	32	110.6
10	92.7	34	113.4
12	93.2	36	113.5
14	94.4	38	116.0
16	95.8	40	116.9
18	97.0	42	117.2
20	98.2	44	113.0
22	100.1	46	94.0
24	101.4		

8. After the distillation was completed, the distilled material was returned to the proper waste bottle and the heat shut off.
9. The apparatus was carefully taken apart and all pieces were returned to the organic chemistry locker.

Figure 2; Boiling temperature (°C) vs volume of distillate (mL) for the Simple Distillation



Discussion

- The temperature in the fractional distillation increased slowly from 82°C to 85°C until the amount of distillate reached 22mL, where there was a dramatic increase to 117°C.
 - This is expected in fractional distillations as before 22mL, the vapour consists of 2-propanol which has a boiling point of 82.6°C (PubChem, 2015). After all of the 2-propanol has been vaporized from the solution, the temperature rose to 117°C which is the boiling point for 1-butanol (PubChem, 2015).
 - The temperature increasing and maintaining 117°C indicated that all of the 2-propanol had been evaporated and that the 1-butanol begins to be evaporated collected.
- In the simple distillation, the temperature slowly increased at a constant pace until approximately 46mL, where there was a dramatic drop in temperature from 113°C to 94°C.
 - An increase throughout the distillation is expected, as the ratio of compounds in the vapour changes throughout the evaporation. In the beginning, more of 2-propanol is evaporated meaning a lower temperature as it has a lower boiling point. As the second compound, 1-butanol, is evaporated, the temperature will increase as it has a higher boiling point than the 2-propanol.
- The fractional column used in the fractional distillation provided a purer product as the added surface area conducted mini-distillations the further separated the 2 compounds when compared to the simple distillation. By looking at Figures 1 & 2 it can be seen that an apparent change in temperature in the fractional distillation marks the transition from the evaporation of 2-propanol to 1-butanol, where the simple distillation has no such immediate increase and a variety of compound ratios evaporating at any given time.
- However, the time required to begin and complete the simple distillation was much quicker than the fractional distillation, while requiring less equipment.

Boiling points taken from PubChem, a database from the U.S National Library of Medicine, last revised 2015. <http://pubchem.ncbi.nlm.nih.gov/search/#collection=compounds>

Questions

1. In a fractional distillation, the inert material within the fractional column provides a surface to which vapour may condense during the distillation. The heat from the constantly rising vapour causes the 2-propanol that has condensed in the column to re-vaporize and eventually be collected. However, the 1-butanol that has been condensed in the column must return back into the distilling flask for a relatively pure end result. The return of the 1-butanol can be difficult as the vapour continues to rise, therefore liquid flowing back through the fractional column will carry any condensed 1-butanol that had been carried into the column, resulting in a purer solution of 2-propanol.
2. A more constant temperature will ensure that all of the lower-boiling compound evaporates without the boiling of the higher boiling compound. If the temperature was to fluctuate, the condensed 2-propanol may drip back into the distilling flask if the temperature is too low or the condensed 1-butanol may evaporate if the temperature is too high, resulting in an impure distillation. Secondly if the heat becomes too high too quickly, liquids can super-heat above their boiling points and vapour might escape through the apparatus, resulting in a final amount lower than the initial amount of distillate.
3. Since the boiling point corresponds to the atmospheric pressure, the vapour pressure of Benzene is 1 atm at 81°C in an open system.
4. The boiling point occurs when the vapour pressure within the liquid is equal to the pressure surrounding the liquid. Therefore, if the pressure surrounding the liquid were to increase, the vapour pressure within the liquid would also need to increase resulting in a higher boiling point. If the atmospheric pressure were to increase, the boiling point would by association increase and visa versa.
5. The cooling water enters at the bottom of the condenser to immediately cool the vapour coming from the heated distillation flask to liquid, which is then emptied into the receiving flask for collection. Secondly, if the water were to flow from the top to the bottom, it would flow straight out of the bottom opening without cooling the inner tube of the condenser.
- 6.

Compound A - Pressure: 350mmHg

- Temperature: 95°C

mol fraction: A = $\frac{3}{4} = 0.75$

mol fraction: B = $\frac{1}{4} = 0.25$

Raoult's Law:

P partial of A = $0.75 \times 350\text{mmHg}$
= 262.5mmHg

Vapour Pressure = $262.5\text{mmHg} + 37.5\text{mmHg}$
= 300mmHg

Compound B - Pressure: 150mmHg

- Temperature: 95°C

P partial of B = $0.25 \times 150\text{mmHg}$
= 37.5mmHg

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

