Organic Chemistry Lab 2: Distillations

Kaitlyn Armstrong - 8544417

Lab Partner: Anne Leslie - 8637629

TA: Yousef Risha

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Procedure

Procedure was followed as per lab manual (CHM1321 Introductory Organic Chemistry Laboratory Manual 2018, Dr. Tony Durst, Dr. Tito Scaiano, Dr. William Ogilvie, and Dr. Alison Flynn, 2018, Exp. 2, p 20 to 27).

Observations

- Simple Distillation
  - Solution was clear and colourless
  - Light boiling of the solution began around 86°C which is also approximately the temperature at which the first drop of distillate was produced
  - The round bottom flask and distillation apparatus leading up to the condenser with cooling sleeve had vapour around the sides

- Fractional Distillation
  - Solution was clear and translucent with a thick meniscus at the top
  - Slowed greatly at 15 mL
  - Temperature reached and hovered around 118°C
  - Dropped quickly when much of the solution was gone

Results

Table 1: Temperatures of distillate at millilitre intervals for the Simple Distillation

Volume (mL)	Temperature (°C)
2	87.4
3	88.2
4	89.0
5	89.8
6	90.1
7	90.6
8	91.7
9	92.7
10	94.0
11	96.3
12	97.8
13	100.5
14	102.5
15	106.6
16	110.2
17	114.2
18	115.6
19	116.3
20	116.4
21	116.5
22	116.6
23	116.6
24	116.6

Table 2: Temperatures of distillate at millilitre intervals for the Fractional Distillation

Volume (mL)	Temperature (°C)
2	82.1
3	82.4
4	82.5
5	82.6
6	82.7
7	83.0
8	83.4
9	84.0
10	84.3
11	84.5
12	85.7
13	88.0
14	88.4
15	118.1
16	118.2
17	118.3
18	118.3
19	118.3
20	118.3
21	118.2
22	117.9
23	115.7
24	79.8

Graphs

Figure 1: Temperature (°C) vs. Volume of Distillate (mL) for the Simple Distillation

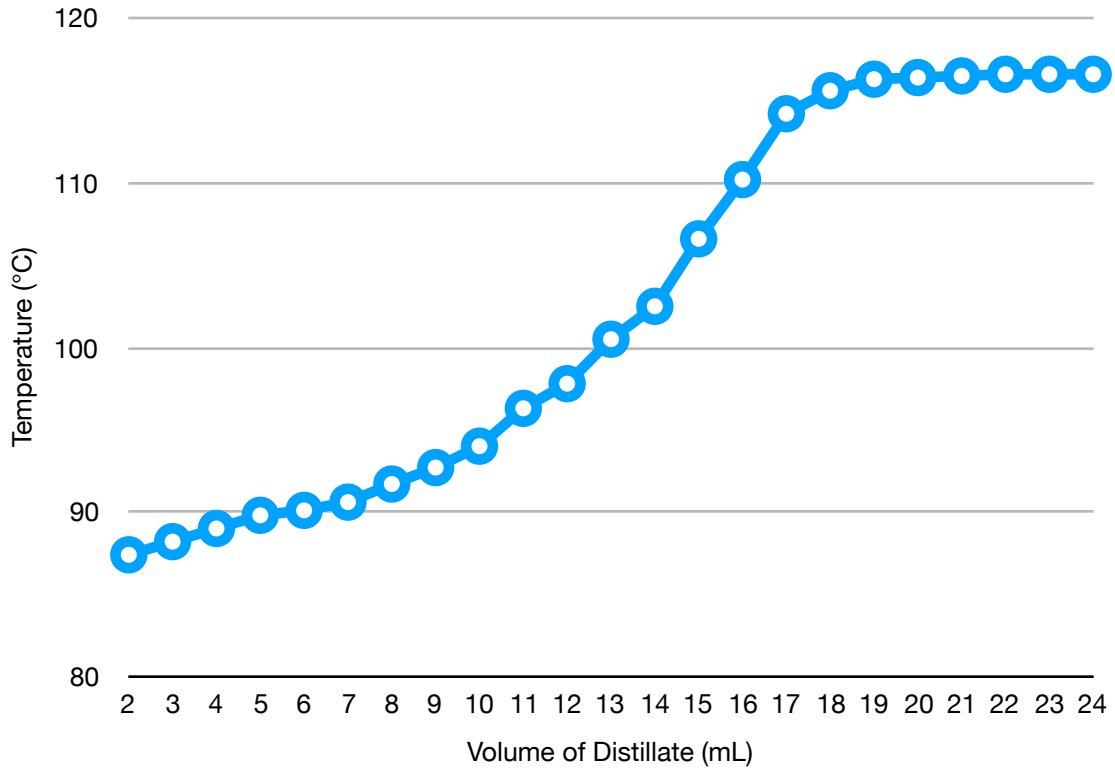
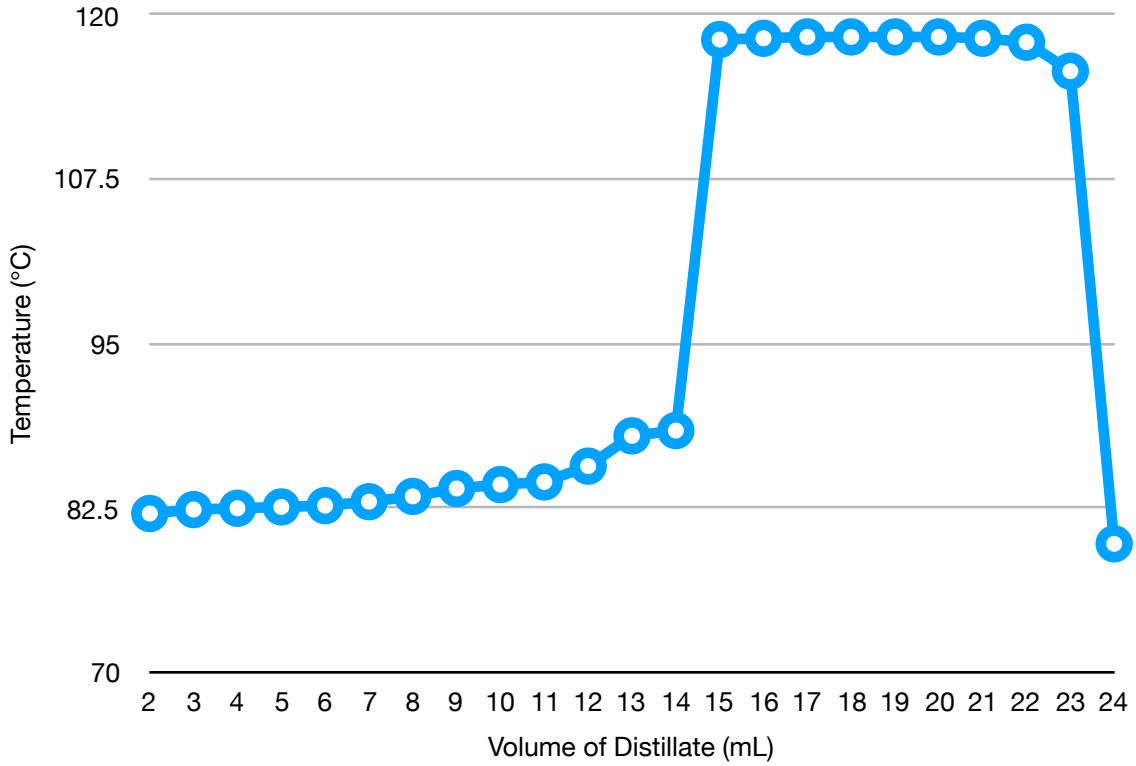


Figure 2: Temperature (°C) vs. Volume of Distillate (mL) for the Fractional Distillation



Discussions

- The goal of the distillations was to separate the 2-propanol from the 1-butanol
- The mechanism of the simple distillation used a distillation head attached to a the condenser allowed for a relatively short distance for the condensation to occur
- This made it easy for both components to travel out regardless of their boiling points
- The difference between the two distillation set ups was in the fractional distillation
- there was a fractioning column in between the round bottom flask and the condenser
- The fractioning column was packed with aluminum shreds to increase the internal surface area
  - allowed for more surface area and length for the vapours to condense on and re-vaporize for a more successful separation
- The condensation and sequential re-evaporation o the vapours in the long fractioning tube resulted in better separation because multiple distillations could occur
  - It would also achieve higher product purity since it uses the multiple distillations process
  - Less efficient because it requires a greater amount of time
  - More effective
- In the simple distillation only a single distillation could occur

- For the temperature vs. volume of distillate for the simple distillation (Figure 1):
  - The curve has a positive slope
  - The slope has the approximate shape of a sigmoid curve
  - This makes sense because more distillate would evaporate as the temperature rose
  - The slope levels out around 118°C, this coincides with the boiling point of the compound with the higher boiling point, 1-butanol (117.7°C)
  - The temperature that 2-propanol requires to vaporize (82.6°C) would have been exceeded, being that it is the lower boiling point compound
  - This coincides with our data because we began to get distillate at approximately 87°C
  - boiling point of a liquid is defined as when the vapour pressure is equal to that of atmospheric pressure
  - The atmospheric pressure of ottawa on January 31st was 102.48 kPa which is only approximately 1 kPa above standard atmospheric pressure meaning boiling point would have been largely unaffected
    - https://www.timeanddate.com/weather/canada/ottawa/historic
  - The graph appears as I would expect with these parameters

- For the temperature vs. volume of distillate for the fractional distillation (Figure 2):
  - The first portion of the graph, between 2 and 14 mL had a slight positive linear slope
  - The second portion of the graph jumped higher on the temperature scale but then had a relatively linear stable pattern, between 15 and 23 mL

- Because of the nature of the fractional distillation and it's ability to separate compound by boiling point, this indicates the boiling points of the two compounds
- The first portion is a curve indicative of the 2-propanol, the compound with the lower known boiling point of 82.6°C
- The second portion is a curve indicative of the 1-butanol, the compound with the higher known boiling point of 117.7°C
- The final point is an outlier
- In an attempt to distill the full volume of the original solution, the experiment was let run and 24 mL was only recorded after the temperature had dropped
- This is likely due to the fact that there was no / very little solution remaining to boil for heat
- The data from the fractional distillation was provided to the class by students Vanessa Levy and Priya Sarwai who were successful in completing the lab experiment
    - Due to the fact that their experiment was preformed beside ours, my observations included were ones I was able to see while also doing our own experiment
    - A full list of their observations is included in raw data
- With atmospheric pressure being approximately standard on this day causing no notable affect in change in boiling point my graphs meet my expectations when disregarding the point at 24mL

- Error
    - My lab partner and I used the graduated cylinder that we'd previously used to collect the 1:1 mixture in to collect the distillate
        - This could mean that if not all the solution was emptied into the round bottomed flask that the resulting distillate was altered in composition or ratio by it's un-distilled form
    - Due to time restraints my lab partner and I were unable to complete the fractional distillation
        - We completed the setup and started the experiment but never got the heat high enough to get any data
    - Human delay in recording temperature as volume of distillate changed millilitres could effect the curvature of the graph
    - The graduated cylinder used began at 2mL so we do not have a 1mL data point to plot for increased accuracy and a closer examination of the boiling point of the compound with the lower boiling point

Questions

1.

- Liquid flow back is to have effective separation of the components of the 1:1 mixture
- The compound that drips back down is that with a higher boiling point - 1-butanol
- It doesn't retain enough energy to make it all the way up the column and into the condenser so it returns to liquid
- The compound with the lower boing point, 2-propanol, is successful in doing so
- This is how they are separated

2.

- Maintaining uniform temperature in the fractioning column allows more distillations to occur
- A drop in temperature would cause a loss in vapours and a return to liquid state
- Temperature fluctuation between the optimal temperature and one outside this range would cause the loss or creation of less vapour
- This makes a less efficient distillation resulting in a less efficient or effective separation

3.

- The boiling point of a liquid is defined as when the vapour pressure is equal to that of atmospheric pressure
- This means the vapour pressure of 81°C benzene is 1.0 atm (or 760 mmHg or 101.325 kPa)

4.

- An increase in atmospheric pressure would increase the boiling point of a liquid
- This is because a boiling point of a liquid is defined as when the vapour pressure is equal to that of atmospheric pressure so it would take more energy to reach

5.

- The condenser's cooling outer layer will be more completely filled when the input of cool water is from the bottom
- If it was input at the top, water would flow down in streams, it would attempt to flow out, and air bubbles would form
- Filling from the bottom allows for a complete fill for more effective cooling

6.

- Composition of compound 3:1
- 75% compound A, pressure 350 mmHg
- 25% compound B, pressure 150 mmHg
- Raoult's law says total vapour pressure is equal to the partial pressure of a pure liquid multiplied by it's mole fraction in the mixture, plus the partial pressure of another pure liquid multiplied by it's mole fraction
- Therefore:

Vap. Pressure Mixture = (mole fraction A)(Vap. Pressure)+(Mole Fraction B)(Vap. Pressure B)
= (0.75)(350mmhg) + (0.25)(150mmHg)
= 262.5 mmHg + 37.5 mmHg
= 300 mmHg

Therefore the vapour pressure of the 3:1 mixture is 300 mmHg

Raw Data - My Own

Jan 31

Lab 2 - Simple Distillation

ml	Temp
2	87.9
3	88.2
4	89.0
5	89.8
6	90.1
7	90.6
8	91.7
9	92.7
10	94.0
11	96.3
12	97.8
13	100.5
14	102.5
15	106.6
16	110.2
17	114.2
18	115.6
19	116.3
20	116.4
21	116.5
22	116.5
23	116.6
24	116.6
25	

Yousef's Tips ☺

- comp w/ lower BP will exit first
- higher bp will remain
- aluminium increases surface area
- highe BP compound will interact with surface area of aluminium
- offers better seperation
- 2 compounds should have a viscosities
- this distilation should be more accurate

- Procedure
  - set up distillation as per lab manual.
  - collected solution.
  - turned on heating pad + stirrer

Raw Data - Other Students

Fractional Distillation @ start 26.2°C

- 2mL - 82.1°C
- 3mL 82.4°C
- 4mL 82.5°C
- 5mL 82.6°C
- 6mL 82.7°C
- 7mL 83.0°C
- 8mL 83.4°C
- 9mL 84.0°C
- 10mL 84.3°C
- 11mL 84.5°C
- 12mL 85.7°C
- 13mL 89.0°C
- 14mL 88.4°C
- 15mL 115.1°C
- 16mL 118.2°C
- 17mL 118.7°C
- 18mL 118.3°C
- 19mL 118.3°C
- 20mL 118.3°C
- 21mL 118.2°C
- 22mL 117.9°C
- 23mL 115.7°C
- 24mL ~~[illegible]~~ 79.8°C

Fractional

↳ begin solution was clear, transparent

↳ thick meniscus at the top

↳ dripped at a rate of 1-2 drops / 5 seconds

@ 11mL, starting to see a (sort of different refraction - looks like viscous compound

↳ taking a very long time

↳ lots of liquid is rapidly dripping back into sample (means good for separation)

↳ at end, almost @ 15mL and when a drop comes, it's like a blob of something more viscous but then spreads down & then to the side

* there unaccounted amount of [illegible] mL went? Aluminum + [illegible]

↳ compound/material is [illegible] every time a drop gets added

↳ not forming a perfectly straight border, more like the curved underside of a meniscus

→ hovering above around above ±7mL

↳ keeps fluctuating b/t 11? 2? 13

↳ going much faster now (dripping)

↳ still dripping quickly @ front of apparatus

↳ glass thing is super super hot

↳ slowing down now as it's approaching 24 mL → still dripping

@ front so [illegible] condensation is where [illegible]