Lab #2

Purifying Chemicals by Distillation

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Introduction:

Distillation is a method of separation of two or more compounds based on the different boiling points they possess. In this experiment, a simple and a fractional distillation were effectively carried out. The aim of this experiment is to understand the concept of distillation and to compare its two types, simple and fractional distillation.

Procedure and observations:

Part 1: Simple distillation

Procedure:

- A simple distillation apparatus was assembled as described in the CHM 1321 Organic Chemistry lab manual (experiment 2).
- The distillation flask was filled with 50 mL of a 50:50 mixture of 2-propanol and 1-butanol using a funnel.
- The 50 mL graduated cylinder was weighted empty. It was 69.5224 g.
- The rest of the setup was done as mentioned in the lab manual (distillation head, condenser, 50 graduated cylinder, thermometer ...)
- A magnetic stirrer was used to stir the solution (not at a maximum speed, just enough to generate a gentle vortex)
- The solution was distilled slowly using minimum heat.
- The temperature was recorded every 2 mL of collected distillate until the distillation was complete.
- After the simple distillation was complete, the cylinder was weighted again containing the distillate. It weighted 107.1386 g.
- Distillate was discarded.

Observation:

- The 50:50 mixture of 2-propanol and 1-butanol was a clear colorless solution with a very strong odour.
- Few minutes after distillation began, few drops of distillate started to drop in the graduated cylinder.
- The initial temperature was 20.8°C.
- After the first 2 mL were collected, the temperature suddenly jumped to 90.3°C and continued to rise gradually as distillate was collected.

Data table

Table 1. Temperature after each 2 mL portion of distillate – Simple distillation

Volume (mL)	Temperature (°C)
0	20.8
2	90.3
4	90.8
6	92
8	92.6
10	93.5
12	94.4
14	95.8
16	96.9
18	98.1
20	99.6
22	101.5
24	103.6
26	105.9
28	107.9
30	110.9
32	112.4
34	114.4
36	116.1
38	116.9
40	117.6
42	118.0
44	118.1
46	118.2

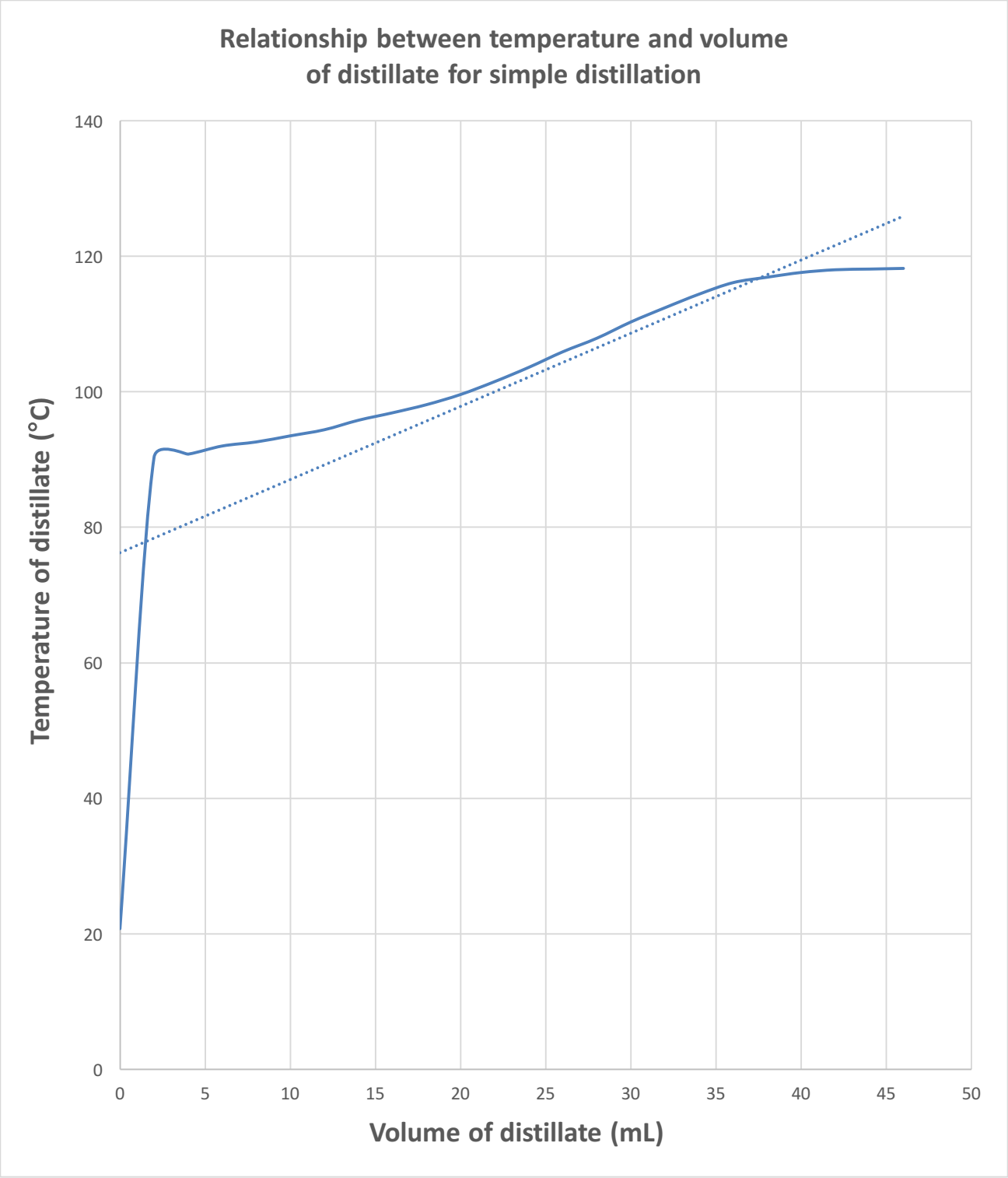


Figure 1. Graph of temperature vs. Volume of distillate for simple distillation.

Part 2: Fractional distillation

Procedure:

- The simple distillation apparatus was partially disassembled as mentioned in the lab manual.
- The distillation flask was filled with 50 mL of a 50:50 mixture of 2-propanol and 1-butanol.
- A fractional distillation apparatus was assembled as described in the lab manual (clamped properly for maximum safety).
- The mixture was distilled slowly and carefully in attempt to avoid flooding in the column.
- The temperature was recorded at each 2 mL of collected distillate until the distillation was complete.
- After the fractional distillation was complete, the cylinder was weighted again containing the distillate. It weighted 106.8148 g.
- Distillate was returned to waste bottle.

Observation:

- Few minutes after distillation began, few drops of distillate started to drop in the graduated cylinder. In this method, it took a little bit longer for the distillate to begin dropping.
- The initial temperature was 21.5°C.
- For the first 2 mL the temperature suddenly jumped to 83.0°C and continued to rise gradually as distillate was collected.

Data table

Table 2. Temperature after each 2 mL portion of distillate – Fractional distillation

Volume (mL)	Temperature (°C)
0	21.5
2	83.0
4	83.8
6	84.3
8	84.6
10	85.1
12	86.0
14	86.5
16	87.5
18	89.0
20	90.8
22	94.7
24	99.6
26	106.5
28	112.5
30	115.2
32	116.7
34	117.6
36	117.9
38	118.0
40	118.1
42	118.1
44	118.1
46	118.1

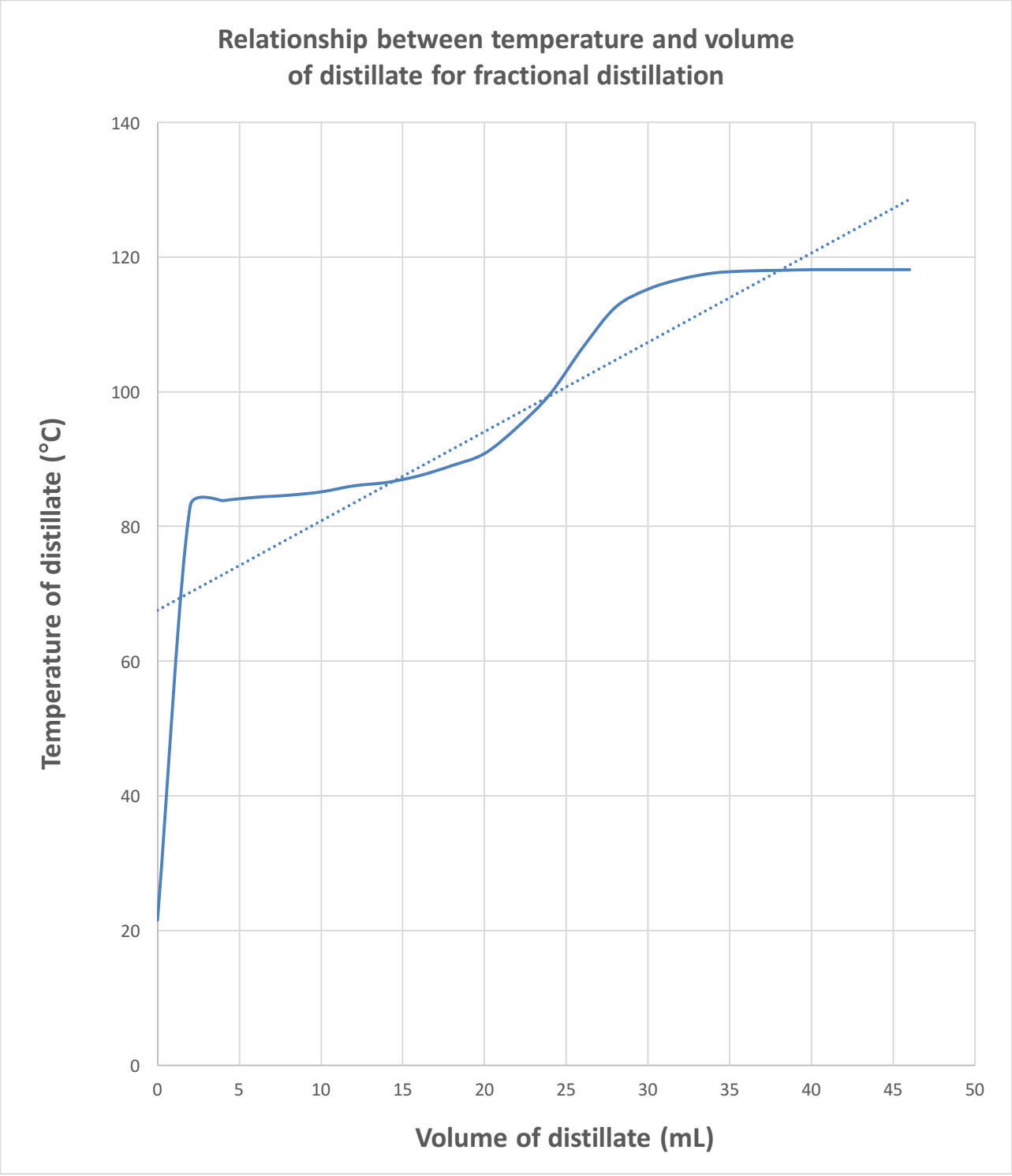


Figure 2. Graph of temperature vs. Volume of distillate for fractional distillation.

Discussion:

Part A. Simple and fractional distillation comparison

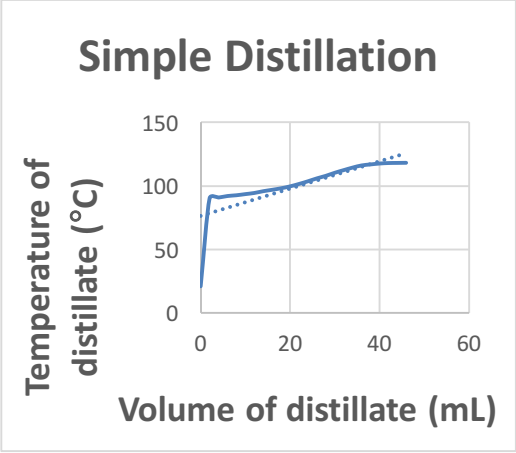
Both simple and fractional distillation require the same equipments: Heating mantle, distillation flask, distillation head, thermometer adaptor, thermometer, condenser, vacuum takeoff adapter and a receiving flask (graduated cylinder). The only difference is that the fractional distillation apparatus has an extra piece called fractionating column.

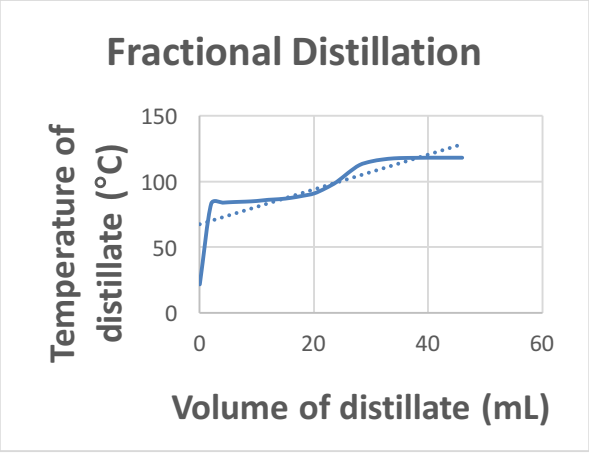
The set up of the distillation apparatus can be considered the most difficult part of the experiment especially that we're dealing with expensive glassware. However, the setup up of the simple distillation apparatus is easier and simpler that the fractional setup. Also, simple distillation is faster and consumes less energy than fractional distillation.

In addition, Fractional distillation gives a better separation between liquids and can purify complex mixture more efficiently than simple distillation.

While simple distillation is best used for the separation of pure liquids with large differences in boiling points, fractional distillation is best used to separate more complex mixture with smaller boiling points differences. The boiling points of 1-butanol and 2-propanol are 117.4°C and 82.6°C respectively.

Part B. Simple and fractional distillation temperature/volume graphs comparison





- In simple distillation, the curve rises suddenly. It then continues on a straight line because there is only one phase (steady process).
- In fractional distillation, the curve also rises suddenly. It then continues to rise gradually because there are several steps (there are times between phases where no change occurs).

- For both graphs, the curve should not rise suddenly for the first 2 mL. The source of error might be that the thermometer was not properly placed at the beginning as negative values were obtained.

Questions:

1) Fractional distillation is a process of separating components via the repetition of vaporization-condensation process (at each cycle it becomes more and more pure). Components with different boiling points will evaporate at different times. The vapor of the component with the higher boiling point condenses in the fractionating column and flows back into the distillation flask and re-boils while the vapor of the component with lower boiling point continues up to the condenser then drops in the receiving flask (volatility).
2) The liquid needs to evaporate and go through the fractionating column before being condensed in the condenser and being collected in the receiving flask, this is easier when the temperature in the fractionating column is maintained uniform. The component can then travel up the column at a certain temperature without having to condense as it reaches a cold spot(top) and drop back.
3) The boiling point of any substance is the temperature at which the vapor pressure of the substance is equal to the atmospheric pressure. So it's equal to 760 torr.
4) The boiling point of liquids increases as the atmospheric pressure increases.
5) If cooling water enters the top of the condenser, the condenser would not fill because gravity will attract water downwards before filling the top and the distillation would not be efficient.
6) Mole fraction of A = 3/ (1 + 3) = 0.75
   Mole fraction of B = 1 - 0.75 = 0.25
   Partial pressure of A= 0.75 x 350 = 262.5 mmHg
   Partial pressure of B = 0.25 x 150 = 37.5 mmHg
   Vapour pressure of the mixture = 262.5 + 37.5 = 300 mmHg.

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Raw Data

Ingrisell?
Nadia
7704594
416-705-1492

* 2-propanol + 1-butanol : (50:50 mixture)

- colorless, transparent, clear
- strong odour.
- 50 mL taken

Simple

* Weight of graduated 50 mL cylinder (empty)

69.5224 g

*

Time	Temp. °C
initial	20.8
2 mL	90.3
4 mL	90.8
6 mL	92
8 mL	92.6
10 mL	93.5
12 mL	94.4
14 mL	95.8
16 mL	96.9
18 mL	98.1
20 mL	99.6
22 mL	101.5
24 mL	103.6
26 mL	105.9
28 mL	107.9
30 mL	110.3
32 mL	112.4
34 mL	114.4
36 mL	116.1
38 mL	116.9
40 mL	117.6
42 mL	118.0
44 mL	118.1
46 mL	118.2
48 mL	→ 47.5 mL : 117.7
50 mL	

HJ

graduated cylinder filled with distillate.

→ 107.1386 g

fractional

Another 50 mL of (50.50) 2-propanol and 1-butanol.

Time (2 mL)	°C
initial	21.5
2 mL	83.0
4 mL	83.8
6 mL	84.3
8 mL	84.6
10 mL	85.1
12 mL	86.0
14 mL	87.5
16 mL	87.5
18 mL	89.0
20 mL	90.8
22 mL	94.7
24 mL	99.6
26 mL	106.5
28 mL	112.5
30 mL	115.2
32 mL	116.7
34 mL	117.6
36 mL	117.9
38 mL	118.0
40 mL	118.1
42 mL	118.1
44 mL	118.1

46 mL	118.1
48 mL	→ 47 mL:
50 mL	118.2 °C

ZD.

Weigh of cyl with d:

→ 106.8148 g.