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Lab Day (T/W/Th/F): W
Lab Week (even/odd): even
Lab time: 10:00

Experiment 2.
Purification of Chemicals through Distillation

Initials: H.P.

Procedure:

Set up

- A paper towel was used to clean the joints of the glassware
- A 25 mL round-bottomed flask was obtained
- 25 mL of 50:50 mixture of 50 mixture of 2- propanol and 1-butanol was added using a graduated cylinder
- A magnetic stirrer was obtained and inserted into the flask
- A magnetic plate was plugged in and a heating mantle was putted on top of the plate
- The flask was clamped using an extension clamp above the magnetic plate
- A jack was placed under the magnetic plate
- The magnetic plate was elevated so that the mantle makes contact with the flask
- A distillation head was attached to the flask
- A temperature adapter was inserted into the top opening of distillation head
- A liebig condenser was attached to the distillation head and secured with a clamp
- One end of a long rubber hose was attached to the bottom of the condenser
- The other end of the rubber hose was attached to the water source
- One end of a shorter rubber hose was attached to the top of the condenser
- The other end of the rubber hose was attached to the water sink
- The metal probe of the thermometer was inserted into the adapter in a manner where the probe is slightly below exit head of the distillation head
- A vacuum take off adapter was attached to the condenser and secured with a C-clamp
- A clean graduated cylinder was placed directly below the vacuum take off adapter
- The thermometer was turned on

Part A: Simple Distillation

- The magnetic plate was turned on to setting 6 and a temperature of 26.7℃
- The mixture was slowly distilled
- The thermometer reading was recorded every time the volume in the graduated cylinder increase by 1 mL
- The setting was adjusted as necessary
- Measurements were made up to 16 mL

Part B: Fractional distillation

- The probe was removed from the adapter
- The still was disassembled
- The mixture in the graduated cylinder was transferred to the flask
- The fractional column was attached to the flask

- The distillation head was attached to the fractional column
- A liebig condenser was re-attached to the distillation head and secured with a clamp
- One end of a long rubber hose was attached to the bottom of the condenser
- The metal probe of the thermometer was inserted into the adapter in a manner where the probe is slightly below exit head of the distillation head
- A vacuum take off adapter was attached to the condenser and secured with a C-clamp
- A clean graduated cylinder was placed directly below the vacuum take off adapter
- The thermometer was turned on
- The magnetic plate was turned on to setting 6 and a 80.3°C
- The mixture was slowly distilled
- The thermometer reading was recorded every time the volume in the graduated cylinder increase by 1 mL
- The setting was adjusted as necessary
- The column was checked for flooding on a regular basis
- Measurements were made up to 20 mL

Observations and Data:

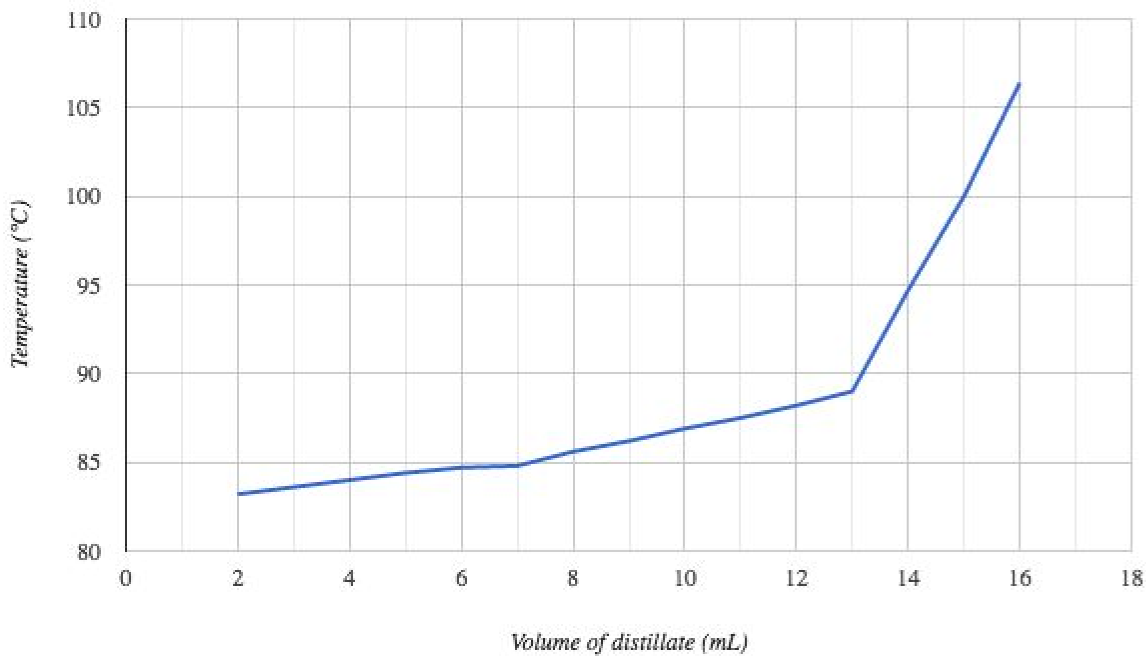
Part A

Table 1. Experimental data of temperature measurement over 1 mL intervals of distillate in simple distillation

Volume of distillate (mL)	Temperature (°C)	Time (s)
2	83.2	956
3	83.6	975
4	84.0	1220
5	84.4	1340
6	84.7	1450
7	84.8	1570
8	85.6	1690
9	86.2	1810
10	86.9	1940
11	87.5	2065
12	88.2	2295
13	89.0	2490
14	94.7	2775
15	100.0	2955
16	106.4	3250

- Condensation had accumulated on the probe
- At the later stages of the distillation, there was a slower rate of drip
- Condensation found on surface of distillation head
- Distillate dripped through gradual accumulation in vacuum take off adapter
- Distillate possibly has a high refractive index as the top of the distillate appears yellow

Figure 1. The change in temperature over 1 mL intervals of distillate in simple distillation



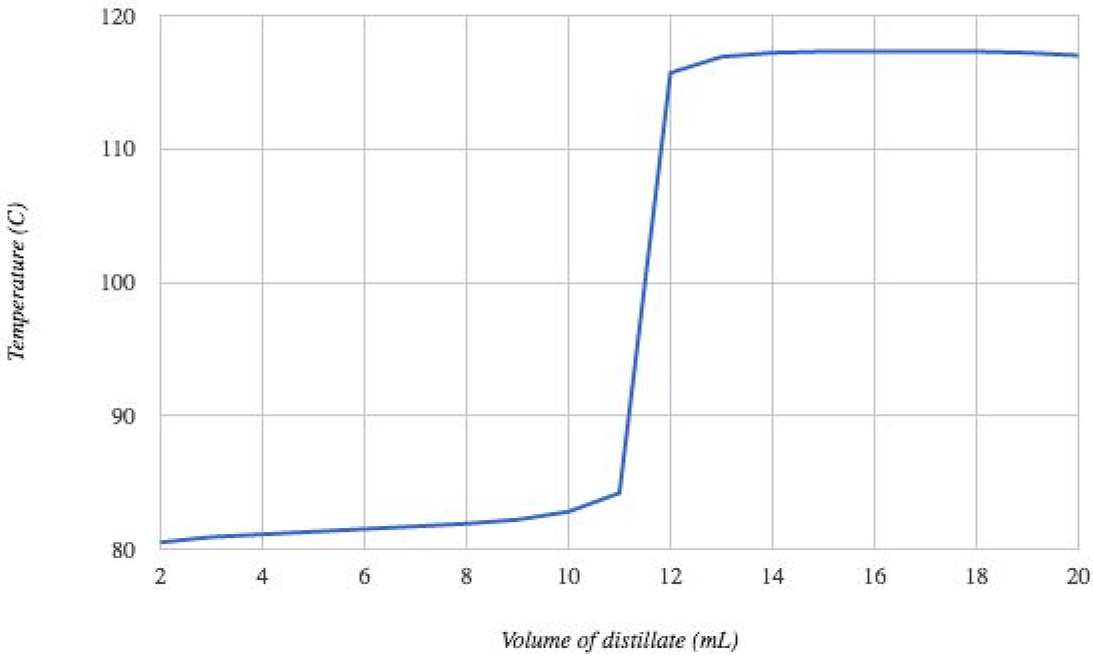
Part B

Table 2. Experimental data of temperature measurement over 1 mL intervals of distillate in fractional distillation

Volume of distillate (mL)	**Temperature (°C)**
2	80.5
3	80.9
4	81.1
5	81.3
6	81.5
7	81.7
8	81.9
9	82.2
10	82.8
11	84.2
12	115.7
13	116.9
14	117.2
15	117.3
16	117.3
17	117.3
18	117.3
19	117.2
20	117

- There was rapid drip-backs into the flask
- Top of distillate appeared yellow (possibly refraction)

Figure 2. The change in temperature over 1 mL intervals of distillate in fractional distillation



Discussion:

The equipment used for simple and fractional distillation were similar except for the addition of a fractional column. This fractional column contained a packing material with a high surface area. This allowed the vapour to condense in the column. This could enhance the process of distillation as some of the condensation may have vapourize (presumably the portion with a low enough boiling point) while the remaining portion drips down to the distilling flask, increasing the number of moles of that chemical in the flask.

The steady increase in measured temperature in the initial stages of the distillations follows Raoult's Law since the distillation causes some of the mixture to vapourize, hence decreasing the mole fraction of the lower boiling-point component while increasing the mole fraction of the other component. As a result, since the Raoult's Law states that the total pressure exerted is the sum of the product of the mole fraction and partial pressure of each component, this decrease in mole fraction for the lower boiling-point component causes the total pressure of the mixture to increase at somewhat of a linear fashion (since the rate of change for the total pressure follows a linear function). Since the total pressure increases at such a rate, the boiling point would increase at a linear rate.

Ideally, in a 50:50 mixture, the large increase in the measured value for the boiling point would be where the volume of distillate is at half of the initial volume of the mixture in the flask (in this case, 12.5 mL). However, both methods of distillation experiences a massive increase in the value of boiling point at volumes of distillate deviates from 12.5 mL. For the fractional distillation, it may be due to intermolecular interactions between the 1-butanol and the 1-propanol where on the packing material, the vaporization of one of the components absorbed some molecules of the other component through polar interactions, assuming that the intermolecular forces between the 1-butanol and the 1-propanol is greater than the intermolecular forces that is causing one of the components to remain as condensation. As for the simple distillation, it is possible that there were droplets of condensed mixture returning back to the distilling flask that was not pure and as a result, some moles of the lower boiling-point substance entered the distilling mixture. The formation of condensation on the bottom of the probe may be factor in this. As a result, it would take longer for the distilling mixture to become pure and experience a massive increase in boiling point. The interval by which the measurements were taken was also a factor since the middle point for the mixture is in between two intervals for measurement. If a graduated cylinder is used as a collection container, the interval should be set so that the middle point is in a value that can be measured or the initial volume for the distilling mixture should be (for the sake of argument) an even number like 20 mL or 30 mL. It may be possible that an impurity entered the mixture, hence exerting partial pressure and affecting the vapour pressure.

The overall technique for both forms of distillation lack difficulty as it only required observations of the apparatus (in the case of fractional distillation, extra observation were required to ensure no flooding occurred). The validity of the temperature readings appear to be sound as the probe's position in the distillation head was enough low enough to ensure envelopment of hot vapour of only one component.

The "purity" of the mixture is very important if one intends to make measurements. If there was additional component in the mixture that one lacks awareness about, this additional component may exert its own partial pressure and cause a deviation from the ideal vapor pressure, affecting the boiling point. Also, considering the alcohol group of the components, there should be polarity in both substances, resulting in a "like-like" interaction and the unlikelihood of positive deviation. However, the longer chain on butanol may contribute towards a hydrophobic characteristic. The resulting products of the distillation appeared to be reasonably pure as the measured temperature of the distilling flask at the later stage of the distillation appears to be consistent towards an approximate 117°C.

One source of error is that for the simple distillation, there was a limited amount of data, so the accuracy of the results is hindered as the boiling point of the component with a higher boiling point cannot be precisely determined than if the entire mixture was distilled. A complete distillation would generate more data that may enhance the precise and accuracy of the result. The instruments used to measure the distillate may also be of issue as the measurement skips 1 mL and begins with 2 mL.

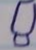
Questions:

1. In fractional distillation, a flowback of condensation into the flask is essential. During the process of fractional distillation, the vapour rises up the fractional column, eventually forming condensation on the packing of the column. Some of the heat of the succeeding rising vapour is transferred to the condensation in the packing. This transferred heat provides enough thermal energy so that one component of the mixture may vapourize but not enough for the other component if the transferred heat is not sufficient for the boiling point of the other component. As a result, the component with the lower boiling point will vapourize while the component with the higher boiling point will remain as condensation. The vapour of the component with the lower boiling point will continue to move into condenser and out onto the graduated cylinder, creating a liquid that is composed of the component with the lower point boiling point. Meanwhile for the other component, the condensation will accumulate in the packing, eventually becoming a size that allows the condensation to flow back into flask. Since this flowback condensation is consist of the substance with a higher boiling point, the flowback will cause the mixture in the flask to be consisted more of that substance than the other. As a whole, this can produce two separate pure liquids.
2. It is important that the fractional column remains in a uniform temperature gradient so that as the vapour moves up the fractional column, the rising vapour may remain as a vapour and exit through the condenser. If the column did not have a uniform gradient of temperature but instead the top portion of the column was at a lower temperature than the bottom portion, then there is the possibility that the component with the lower boiling point may form condensation, which may drip back into the flask. This would result in the substance in the flask to become less pure.
3. Since the boiling point is defined as the temperature at which the vapour pressure of a substance and the pressure that is applied on the substance are equal, the vapour pressure is equal to the atmospheric pressure of the environment, which is 760 torr or 101.1 kPa
4. Considering that the boiling point is related to the atmospheric pressure by acting as an opposing force that is applied against the vapour pressure of the substance, a substance may vapourize if the vapour pressure of the substance overcomes the atmospheric pressure applied on it, allowing for volumetric expansion into a gas. If there is an increased atmospheric pressure, then the vapour pressure of the substance must be greater into order to overcome the increased atmospheric pressure in order to become a gas. As a result, more energy is required to excite the molecules of the substance into order to exert enough force to produce a vapour pressure that is equal to the atmospheric pressure, meaning that the boiling point will increase.
5. By having the cooling water enter the condenser through the bottom instead of the top, it will ensure that the water around the condenser will remain at a cool temperature. As the distillate enters the condenser, the heat of it will transfer to the water nearby. This heated

water, if the water was designated to enter through the bottom, can exited through the top of the condenser and into the sink, ensuring that the heat from that water does not cause the overall temperature of the cooling water to increase dramatically. If the cooling water had enter through the top, then there will be immediate exposure to the heat of the distillate and in order to exit the condenser, the water would have to move down the entire condenser, transferring its heat to the surrounding portions of water and hence, would likely increase the temperature and reduce its effectiveness of a cooling agent.

6. $P_{total} = P_A + P_B$
   $P_{total} = (P_A{}^\circ)(N_A) + (P_B{}^\circ)(N_B)$ = (350 mm Hg)(0.75) + (150 mm Hg)(0.25) = (262.5) + (37.5) = 300 mm Hg
   The vapour pressure of a 3:1 mixture of A and B at 95 °C is 300 mm Hg.

25 mL of 50:50 mixture

- Obtain flask; stirring; clamp (2); jack; ;  ✓therm. tube; Condenser; C-clamps
  3-clamp; Tubing
  sink, source

Cy

condensation on therm.

T_1 = 26.7°C

Distillation

2 mL	: 83.2°C	15:56
3 mL	: 83.6°C	16:15
4 mL	: 84.0°C	20 min 20 s
5 mL	: 84.4°C	22 20
6 mL	: 84.7°C	24 15
7 mL	: 84.8°C	26 10
8 mL	: 85.6°C	28 10
mL → 9	86.2°C	30 10
10	86.9°C	32 30
11	87.5°C	34 25
12	88.2°C	38 15
13	89.0°C	41 30
14	94.7°C	46 15
15	100°C	49 15
16	106.4°C	54 10
17		
18		
19		
20		

Slower rate of flow/drip

T_1 = ~~29.8°C~~ 26.5°C

Fract.

Rapid drip back

#	
2	30°C
3	81.4°C
4	81.7°C
5	81.4°C
6	81.3°C
7	81.1°C
8	80.4°C
9	79.0°C
11	75.4°C

PEN

Fractional distillation

initial temp: ~~33°C~~	Volume	temperature
80.3°	1 mL	~~80.5~~ ~~80.5~~ .
	2	~~80.9~~ 80.5
	3	~~8~~ 80.7
	4	81.1
	5	81.3
	6	81.5
	7	81.7
	8	81.9
	9	82.2
	10	82.8
	11	84.2

* dripping stopped → changed temp to 80 to mediate

12	~~*~~ 115.7	
13	116.9	
14	117.2	
15	117.3	
16	117.3	almost nothing left
17	117.3	almost nothing left
18	117.3	almost nothing left
19	117.2	almost nothing left
20	117.	almost nothing left