LAB 2:

PURIFYING CHEMICALS BY DISTILLATION

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CHM1321 - Z05

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Procedure

See lab manual <u>Experiment 2: Purifying Chemicals By Distillation</u> (pg 1-9)

Qualitative Observations

- A 50:50 dichloromethane:ethyl acetate solution was to be distilled.
- The 50:50 solution was colourless, transparent and had a mildly sweet odor.
- During the collection of the distillate, the meniscus would shake in the graduated cylinder when the drops would fall from the vacuum takeoff adapter. This made it difficult to read the amount of millimeters of distillate collected and could be considered a source of error.
- The pressure at which the distillation was performed is equal to the atmospheric pressure.
- Solution was stirring smoothly for a few minutes and once the heating pad started to raise the temperature there was a gentle bubbling and vortex of the solution in the flask throughout both the simple and fractional distillation.
- Stir setting was kept at a minimum of 3 and a maximum of 5
- For the second part of the lab a fractional distillation was performed. In order to do this a tube with packing was introduced to the apparatus setup from the simple distillation.
- The packing was made of some sort of metal and it had a silver colour.
- The rate of collection for the fractional distillation was much slower than that of the simple distillation which was to be expected.
- During the fractional distillation, while some of the solution was dripping into the receiving flask, a lot more of the solution was seen dripping back into the distilling flask.

Quantitative Observations and Graphs

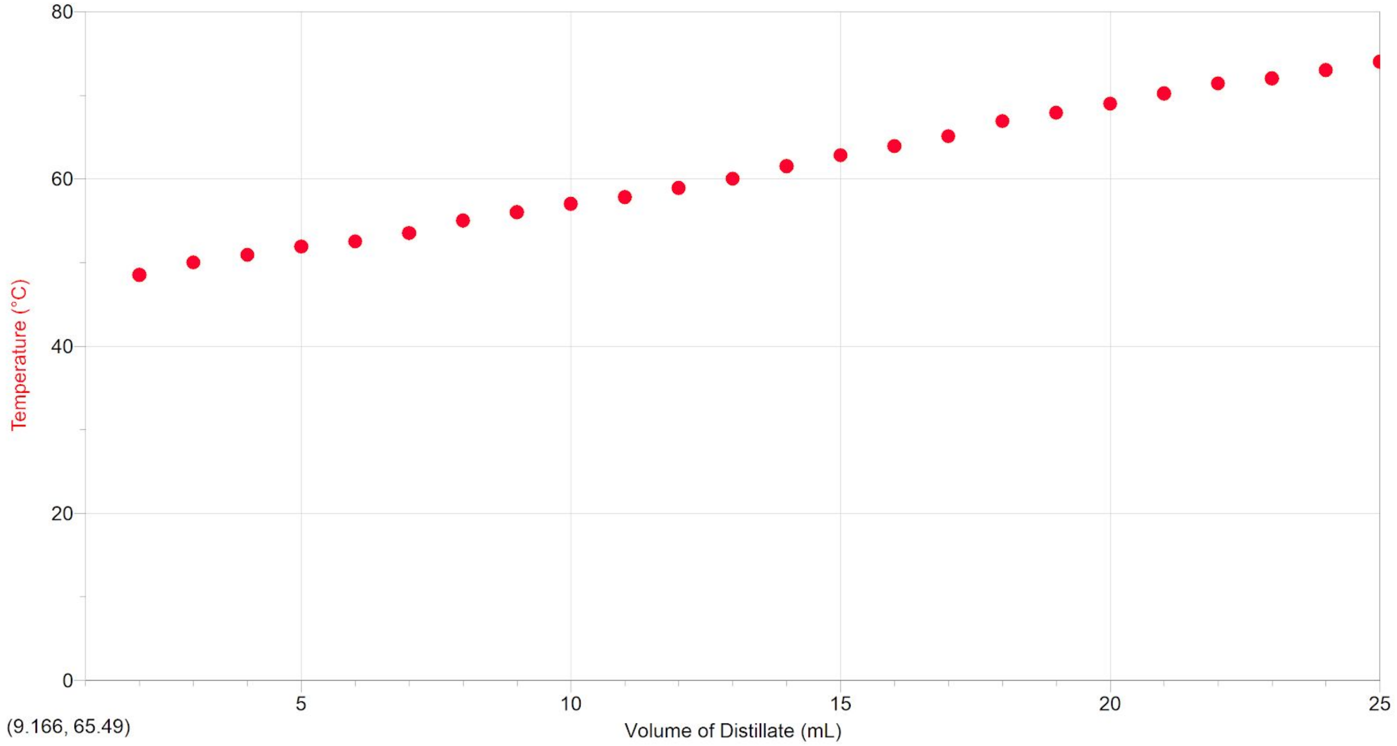
Simple Distillation Observations

Table 1.0 - Collected Data of Simple Distillation: Temperature vs Volume

Volume of Distillate (mL)	0*	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25
Temperature (℃)	21.0*	48.5	50.0	50.9	47.9	52.5	53.5	55.0	56.0	57.0	57.8	58.9	60.0	61.5	62.8	63.9	65.1	66.9	67.9	69.0	70.2	71.4	72.0	73.0	74.0

*This point was recorded before the start of the distillation, thus it does not fit the trend with the rest of the data points collected.

Fig. 1.0. Temperature vs. Volume graph during the simple distillation of the 50:50 solution of dichloromethane and ethyl acetate



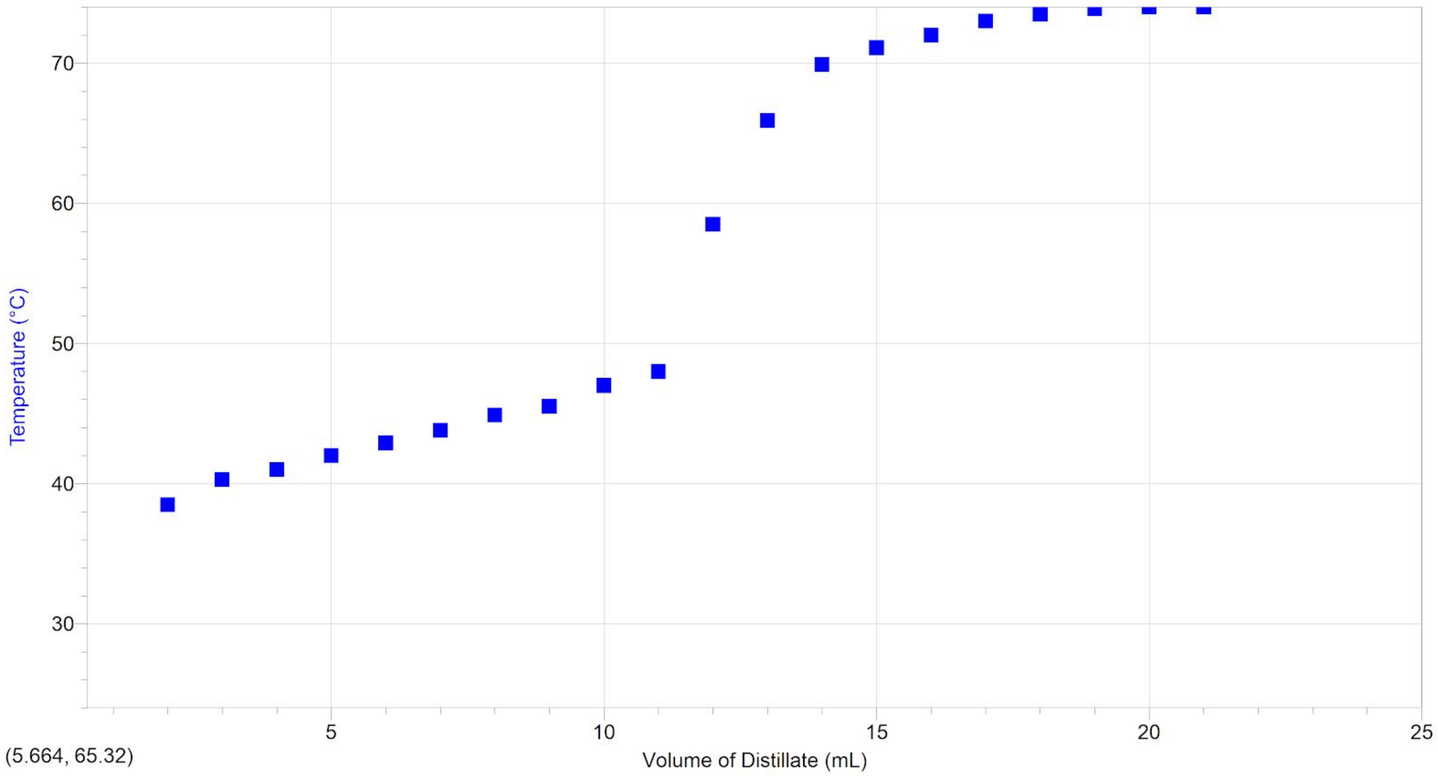
Fractional Distillation Observations

Table 2.0 - Collected Data of Fractional Distillation: Temperature vs Volume

Volume of Distillate (mL)	~~0~~*	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22*
Temperature (℃)	~~25.0~~*	38.5	40.3	41.0	42.0	42.9	43.8	44.9	45.5	47.0	48.0	58.5	65.9	69.9	71.1	72.0	73.0	73.5	73.9	74.0	74.0	~~71~~*

*The first point was recorded before the start of the distillation, thus it does not fit the trend with the rest of the data points collected. The last point was collected at the very end of the distillation when there was next to no solution and the heat was removed.

Fig. 2.0. Temperature vs. Volume graph during the fractional distillation of the 50:50 solution of dichloromethane and ethyl acetate



Discussion

Simple distillation

A simple distillation is a very quick procedure; however, it is best used when the boiling point difference between the two compounds within the solution is over 100°C (“Experiment 2 : Purifying Chemicals By Distillation,” 2014). The compounds in the 50:50 solution for this experiment were dichloromethane and ethyl acetate. The boiling point for dichloromethane is 39.6°C and for ethyl acetate was 77.1°C. The temperature difference between these is 37.5°C much less than the 100° C that is required for an ideal simple distillation. During the simple distillation, the rate at which the temperature increased was linear. Thus, in the graph there was no distinct curve. This can be attributed to the fact that there was little difference between the boiling points of the two components of the mixture. Therefore the graph of the results in figure 1.0. is as expected, proving that simple distillation was not the ideal method for separating the components of this mixture.

Fractional distillation

Fractional distillation was the ideal method in order to separate the two components of the mixture as fractional distillations are used to separate substances with a temperature difference of less than 100°C (“Experiment 2 : Purifying Chemicals By Distillation,” 2014). The compounds in the 50:50 solution for this experiment were dichloromethane and ethyl acetate. The temperature difference in boiling points between these is 37.5°C. During the simple distillation the vapours formed were not pure and did not yield any result showing the difference in boiling points in the two components when graphed. In fractional distillation a fractionating column is used (“Experiment 2 : Purifying Chemicals By Distillation,” 2014). The column consists of a number of obstructions (metal packing) due to which the cooling area for the hot vapours increase (the surface area of the column is increased). When the vapours of both liquids rise in the column, they come into contact with the obstructions over a large area and condense. Vapours of the component having the lower boiling point rise up the column and re-vaporize the lower boiling component condensate in the fractioning column and passes over into the condenser. Whereas, the vapours of the component having the higher boiling point are condensed in the column itself and the condensed liquid flows back into the distilling flask c. Thus, automatically the separation of the two vapours takes place in the column. In figure 2.0, the graph accurately shows that half way through the distillation process the temperature had to raise significantly until distillate was being collected again. As mentioned before the boiling point difference between the two components was 37.5°C which can be seen in the graph. At 2 ml the temperature was 38.5°C and half way through after the temperature increase the temperature was 74.0°C. This means during the experiment the temperature had to rise 35.5°C until the second component was collected as distillate. Which is rather close to the theoretical difference of 37.5°C. Figure 2.0 accurately represented the expected results of a successful fractional distillation showing the classic distillation curve.

Sources of Error

A few sources of error may have hindered the success of this experiment.

A technical source of error was the quality of the equipment. Upon inspection of the apparatus there was a miniscule fracture in the glass at the connection point between the distilling flask and the condenser, some vapour could have escaped here and partially could have been responsible for having less distillate collected than initially having started with. This could have been fixed by using newer or less damaged glassware.

There was an experimental human error midway through the fractional distillation (around 12mL in) there was a spill from the outlet onto the fume hood bottom. In the midst of cleaning the spill someone may have thought that the outlet was in the inlet and plugged in the outlet on accident. This caused flooding on the end of the flask and for a couple of minutes no distillate was being collected and temperature dropped, potentially skewing the results and the distillation curve in the figure 2.0.

It was observed that the final amount of the mixture was slightly short of the 25mL present initially. This is attributed to the fact that dichloromethane evaporates very quickly even at room temperature and while the fractional column was being set up it was left in the graduated cylinder out in the open for at least half an hour. This could have been prevented by covering the graduated cylinder or keeping the whole mixture in a cooler environment while setting up the fractional column.

Correcting the errors mentioned above would definitely improve the distillation curves if the lab were repeated. Although, another thing to consider would be to choose different components. Dichloromethane and ethyl acetate have a rather small difference in boiling point, perhaps choosing components with a difference larger would yield better graphs.

Conclusion

The simple distillation and fractional distillation both yielded results that could have been expected from a 50:50 solution of dichloromethane and ethyl acetate. Due to the boiling point difference being 37.5° C, the simple distillation graph (figure 1.0) turned out to be linear showing no separation between the two components. While the fractional distillation graph (figure 2.0) showed a clear temperature increase between collecting dichloromethane and ethyl acetate. It is also concluded that fractional distillation would have been more helpful if the goal of the experiment was to separate the two components.

Questions

1.

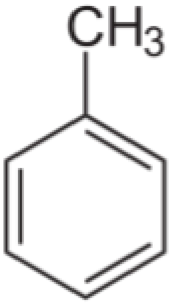


Fig 3.0. Toluene

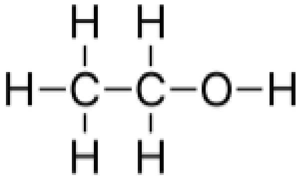


Fig.4.0 Ethanol

- The boiling point of toluene is 110.6°C (National Center for Biotechnology Information. PubChem Compound Database; CID=1140).
- The boiling point of ethanol is 78.2°C (National Center for Biotechnology Information. PubChem Compound Database; CID=702).
- The difference in these boiling points is relatively minimal. $\Delta T = 110.6°C - 78.2°C = 32.4$ C.
- This means that it would be very hard to use simple distillation in order to separate this mixture. In order to perform a successful simple distillation the difference in boiling points needs to be greater than 100°C ("Experiment 2 : Purifying Chemicals By Distillation," 2014)
- Another method that would yield a more successful separation is fractional distillation. In this scenario, there are many mini distillations taking place over one whole experiment which allows more of the lower boiling component to vaporize while the higher boiling component condenses inside the fractionating column and does not join the lower boiling component in the receiving flask, thus enabling better separation ("Experiment 2 : Purifying Chemicals By Distillation," 2014).
- The mixture of toluene and ethanol is also considered to be a minimum boiling azeotropic mixture like that of water and ethanol (Zhu, Z., 2015). Thus, it is not possible to fully isolate a component of this mixture.
- Similar to the way that the azeotropic mixture of water and ethanol is distilled, by the addition of benzene to the mixture, the azeotropic mixture of toluene and ethanol could also be completely separated ("Experiment 2 : Purifying Chemicals By Distillation," 2014).

2.

- As the liquid molecules were vaporized they were transported through the column to the condenser where they resumed to a liquid state and were collected.
- If the temperature is not consistent in the column, the separation will not be as smooth.

- The entire procedure works merely due to the fact that the two components of the mixture have different boiling points and therefore at different temperatures they can be separated.
- If temperature is not on a steady climb and drops at times the distillation will be inefficient.
- The distillate will vaporize and then condense within the distilling flask and fractional column instead of condensing in the receiving flask making the process inefficient.
- Therefore it is important to maintain a smooth temperature gradient in the column

3.

It is given that the boiling point of dichloromethane = 39.6°C.

For any substance, that is a liquid, the temperature at which it is able to boil (the boiling point) is the temperature at which the vapour pressure of that substance is equal to the atmospheric pressure. Therefore, the vapor pressure of dichloromethane or any other substance at boiling point would be 1 atm or 101.3 kPa (Openstax, 2018).

4.

- Boiling occurs when the molecules in a liquid phase have enough energy and volume to spread out and form bubbles.
- The bubbles rise to the surface and release vapourized solvent.
- The more pressure, the less volume for the molecules to escape to go into a gaseous phase.
- When temperature increases molecules move more and spread out more.
- When pressure increases on a liquid, boiling is more difficult and for boiling to occur the temperature must also increase to compensate.
    - This can be explained via Chatelier's principle
- Therefore if atmospheric pressure is increased the boiling point will also increase

5.

Fig.5.0. *Inlet superior to outlet*

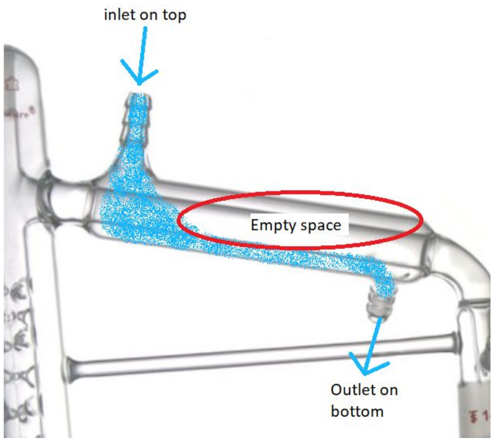
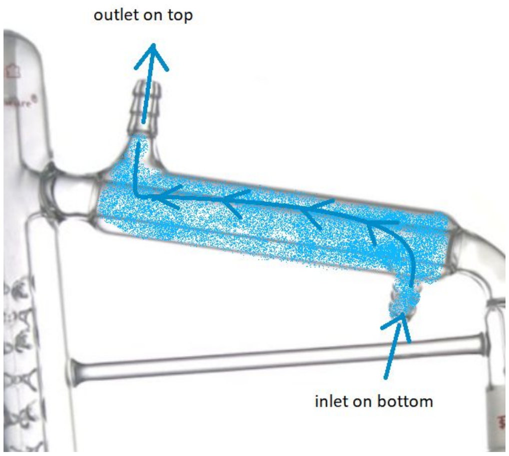


Fig. 6.0*. Inlet inferior to outlet*



- ➢ The purpose of the condenser is to cool the vapour enough to make it go back to a liquid phase, hence the name condenser.
- ➢ To do this, cold water is constantly flowing out and in of the condenser. The condenser needs to be filled with water at all times in order to cool down the vapour and if the water flowed from the top to the bottom there is a chance for the rate of the water flowing out of the condenser to be much larger compared to the rate of water flowing into the condenser.
- ➢ This would mean that some of the condenser jacket would not be filled with water and the overall temperature of the condenser would not be cool enough to condense the vapour so that it may be distilled into the receiving flask.
- ➢ By allowing the water to flow from the bottom to the top, this would mean that the entire volume of the condenser jacket must be filled before the water is to flow out of the condenser and the condenser would always be filled with water to allow for the cooling of the vapour.

6.

Given:

Compound A	Compound B
vapour pressure = 350mm Hg at 95°C	vapour pressure = 140mm Hg at 95°C

A and B are miscible

4:1 Mixture of and A and B

Required:
Vapour pressure of 4:1 mixture of A and B at 95°C

Raoult's law:
$P_{total} = (P_A^{\circ})(N_A) + (P_B^{\circ})(N_B)$

Vapour Pressure =

= (350mmHg)(4/5) + (150mm)(1/5)
= 280 mm Hg + 30 mm Hg
= 310 mm Hg

∴ **Vapour pressure is 310 mm Hg for a 4:1 mixture of A and B at 95°C**

References

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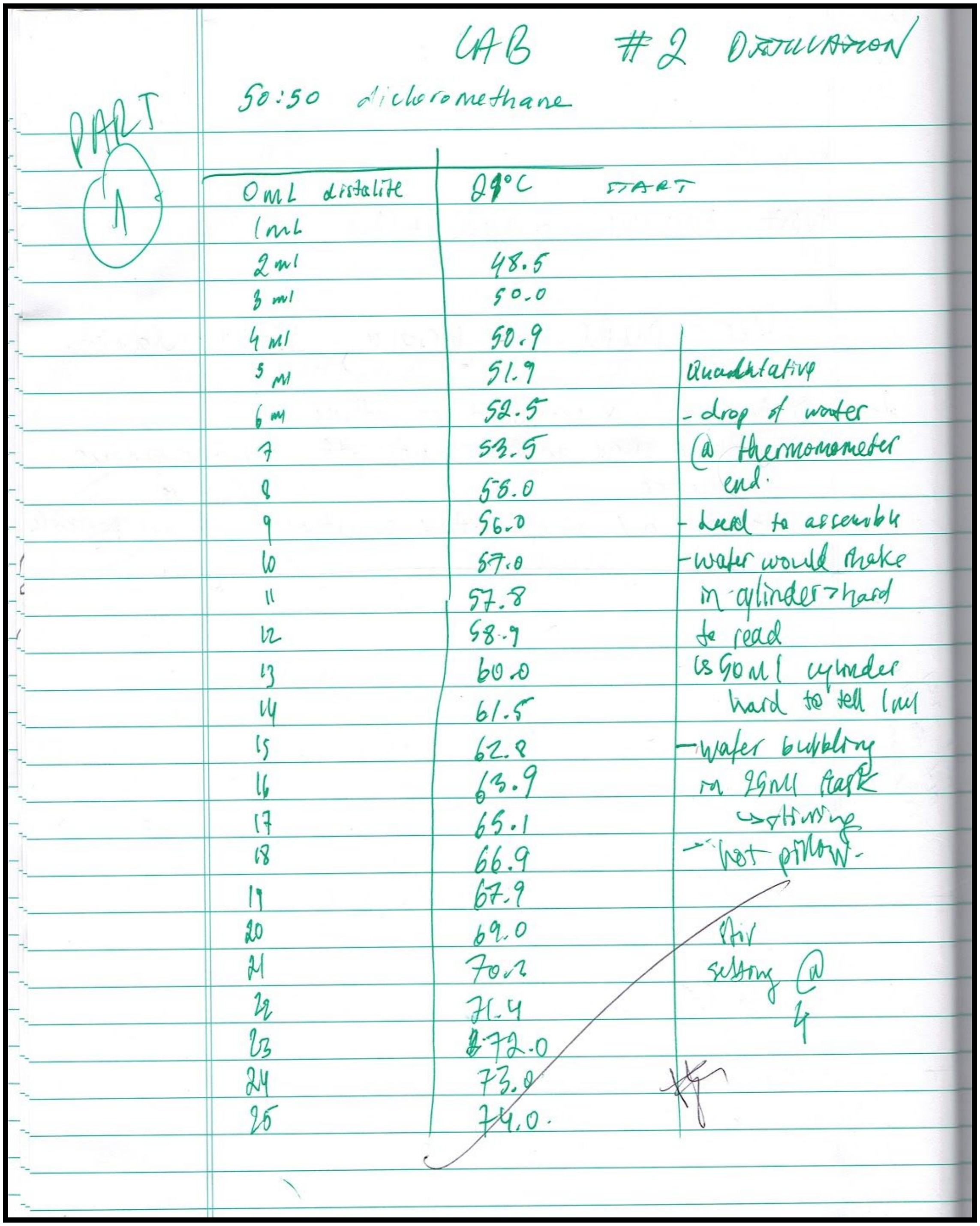


LAB #2 DISTILLATION

PART 1

50:50 dichloromethane

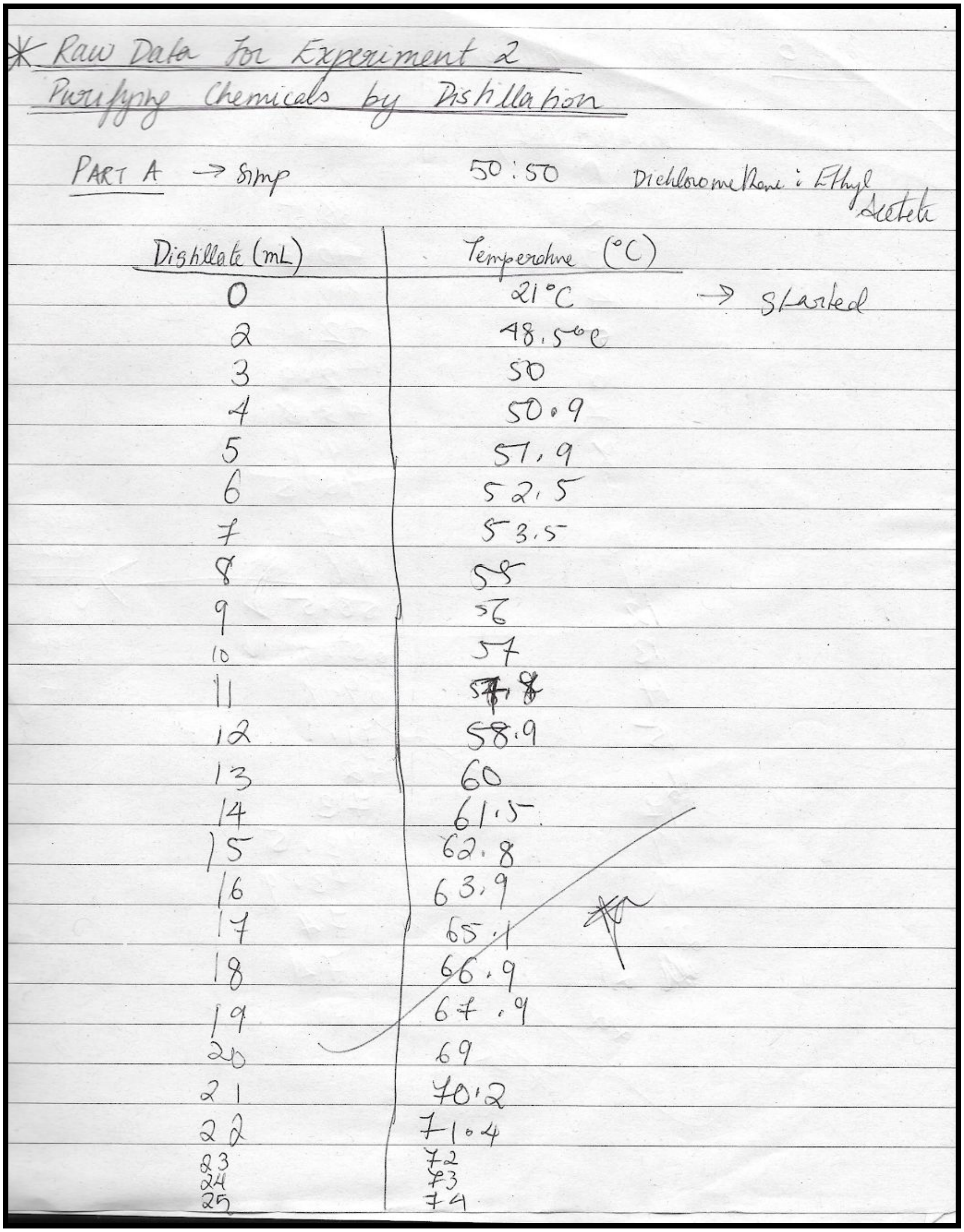
0 mL distalite	23°C	START
1 mL		
2 ml	48.5	
3 ml	50.0	
4 ml	50.9	
5 ml	51.9	Quadlitative
6 ml	52.5	- drop of water
7	53.5	@ thermomometer end.
8	55.0	
9	56.0	- hard to assemble
10	57.0	- water would shake
11	57.8	in cylinder > hard
12	58.9	to read
13	60.0	↳ 50 ml cylinder
14	61.5	hard to tell 1ml
15	62.8	- water bubbling
16	63.9	in 25ml flask
17	65.1	↳ stirring
18	66.9	- hot pillow -
19	67.9	
20	69.0	stir
21	70.2	setting @
22	71.4	4
23	72.0	
24	73.0	
25	74.0.	

little drop of water
@ end of thermometer

PART ②. Fractal Distillation.

ml distallate	Temp °C	
1ml		
2ml	38.5	
3ml	40.3	
4	41.0	
5	42.0	
6	42.9	
7	33.8	
8	44.9	
9	45.5	
10	47.0	
11	48.0.	dropping
12	58.5	@ wrong end
13	65.9	temp went down
14	69.9	
15	71.1	
16	72.0	
17	73.0	
18	73.5	
19	73.9	
20	74.0	~~77~~
21	74.0.	moved away
22	71.1 ?	pillow heat
23		
24		
25		

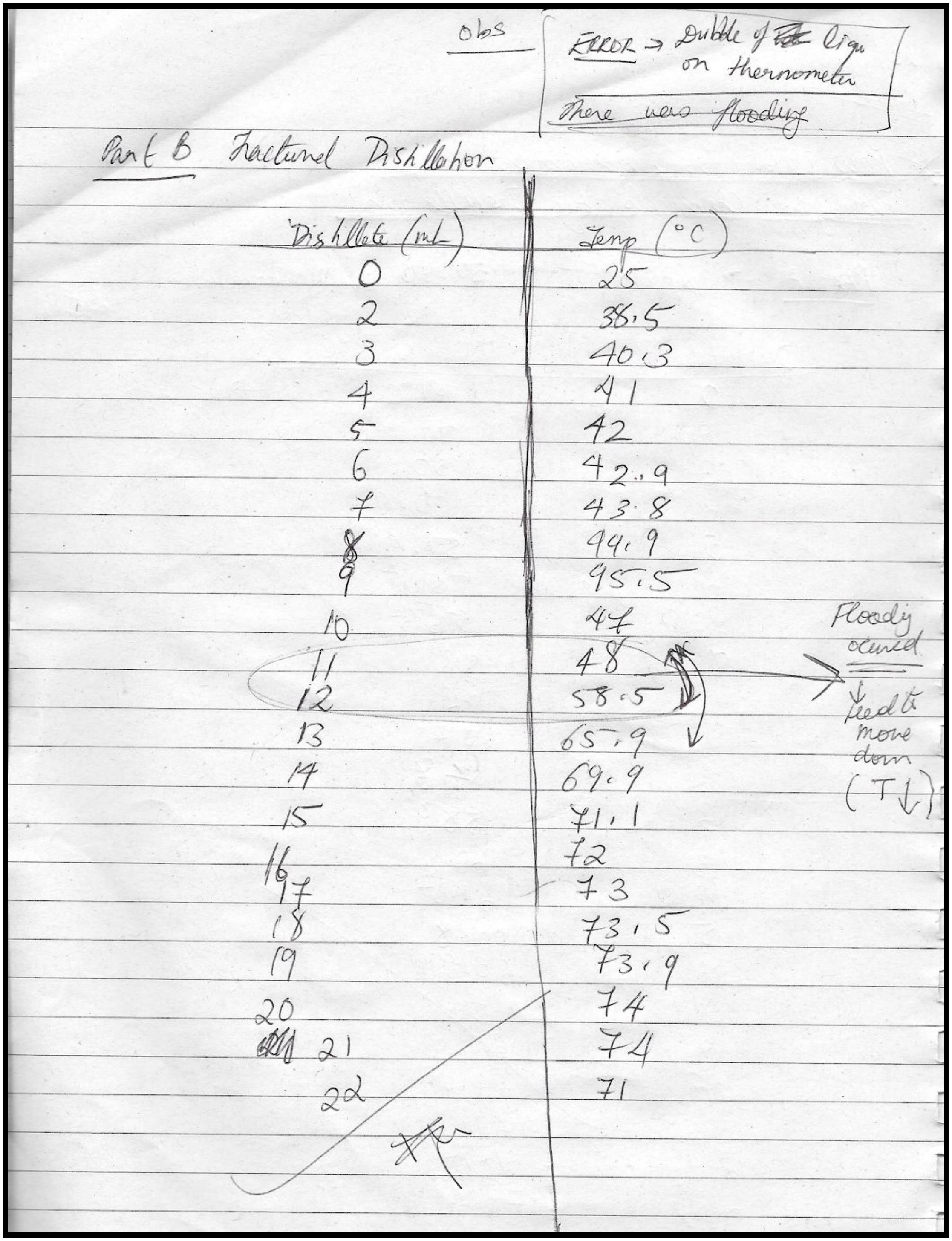
[redacted]



* Raw Data for Experiment 2
Purifying Chemicals by Distillation

PART A → Simp 50 : 50 Dichloromethane : Ethyl Acetate

Distillate (mL)	Temperature (°C)	
0	21°C	→ Started
2	48.5°C	
3	50	
4	50.9	
5	51.9	
6	52.5	
7	53.5	
8	55	
9	56	
10	57	
11	57.8	
12	58.9	
13	60	
14	61.5	
15	62.8	
16	63.9	
17	65.1	
18	66.9	
19	67.9	
20	69	
21	70.2	
22	71.4	
23	72	
24	73	
25	74	



Obs

ERROR → Bubble of ~~[illegible]~~ liqu on thermometer

There was flooding

Part B Fractional Distillation

Distillate (mL)	Temp (°C)
0	25
2	38.5
3	40.3
4	41
5	42
6	42.9
7	43.8
8	44.9
9	45.5
10	47
11	48
12	58.5
13	65.9
14	69.9
15	71.1
16	72
17	73
18	73.5
19	73.9
20	74
21	74
22	71

Flooding occured ↓ lead to move down (T↓)