Experiment Two: Distillation

Section: B01
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Procedure

Refer to procedure for experiment two CHM 1321 Organic Chemistry Laboratory Manual, pages 20-27.
Some modifications were made to the procedure, as listed.

- The 100 mL distilling flask and fractioning column were wrapped in tinfoil for the fractional distillation process.
- The voltage of the heating plate was set to 85 volts, instead of the recommended 65 volts.

Reagents

compound	Amount (mL)
50:50 mixture 2-propanol & 1-butanol	25 mL

Observations

- Solution was a clear liquid
- Solution had pungent, alcoholic smell
- During the distillation, gas could be seen rising through to column and condensing into liquid in the condenser
- About two-thirds of the way through the distillation, the receiving flask clearly had another chemical entering is, as differences in density of the liquid could be seen. (another, also clear liquid interacted with the liquid already in the flask)
- The temperature at the start remained about the same for approximately 10 minutes, (initial temperature of about 25 degrees) before rapidly climbing about 60 degrees in the span of 7-10 seconds.
- After the initial temperature climb, the temperature gradually increased over the course of about 15-20 minutes before the distillation ended.

Data Tables

Simple Distillation
Initial temperature: 24.7 C
Final Temperature: 81.6 C

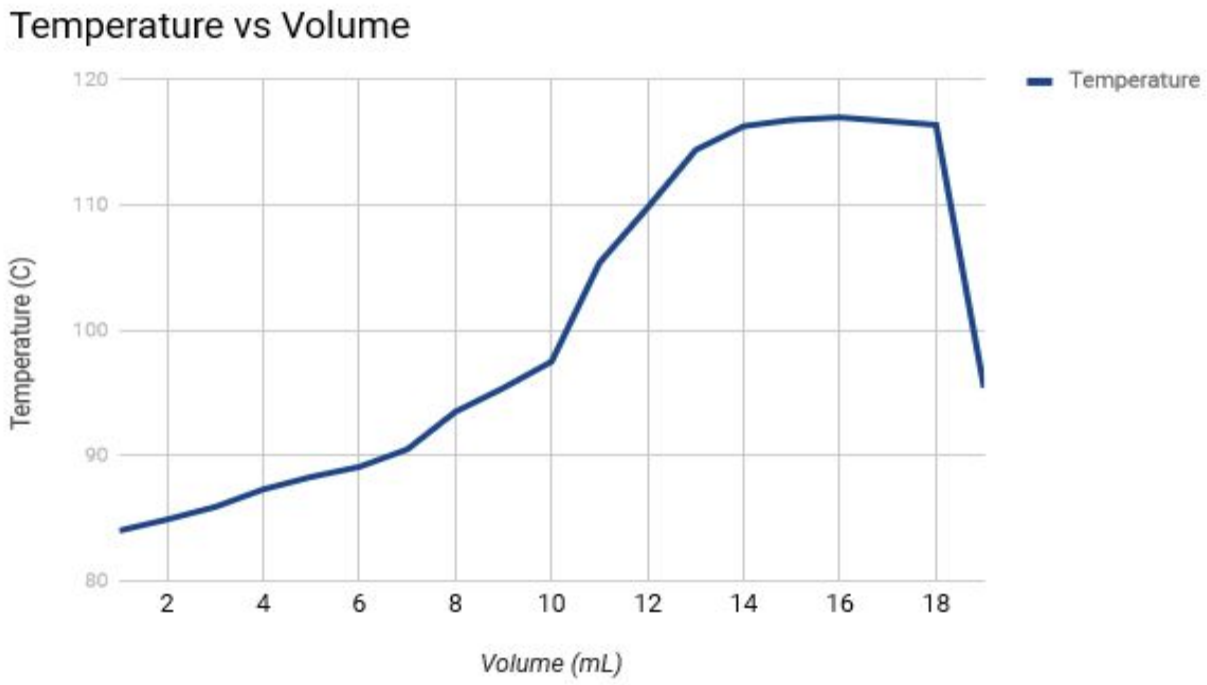
Temperature (degrees C)	Volume in receiving flask (mL)
84.0	1
84.9	2
85.9	3
87.3	4
88.3	5
89.1	6
90.5	7
93.5	8
95.5	9
97.5	10
105.4	11
109.8	12
114.4	13
116.3	14
116.8	15
117.0	16
116.7	17
116.4	18
95.4	19

Fractional Distillation

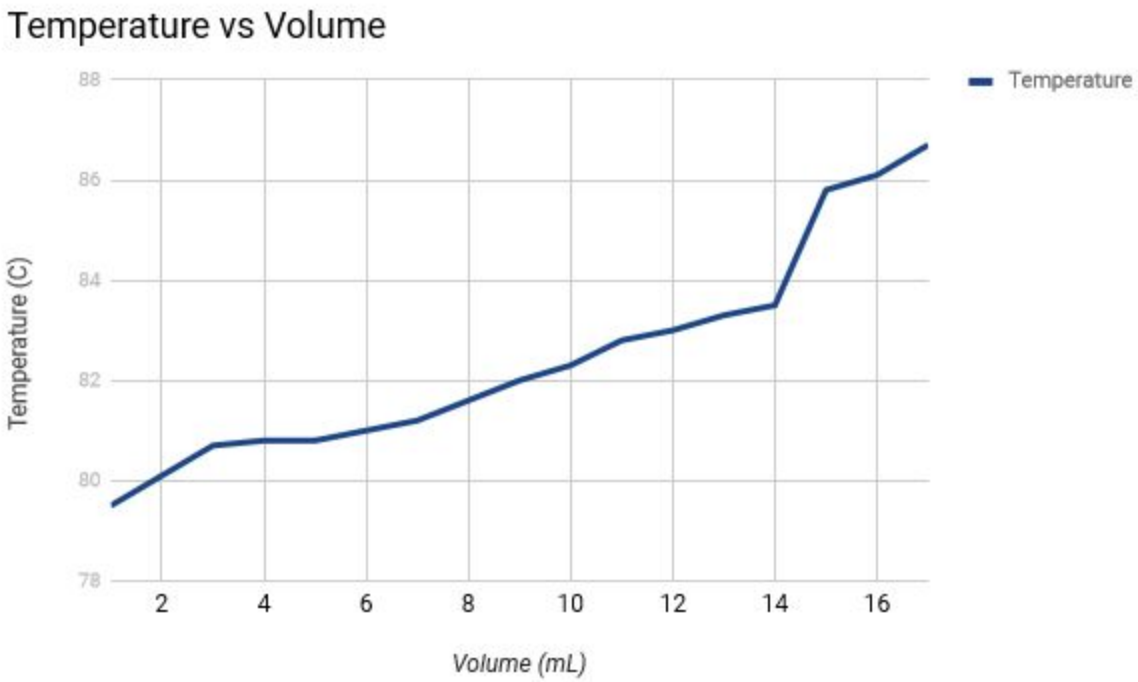
Temperature (degrees C)	Volume (mL)
79.5	1
80.1	2
80.7	3
80.8	4
80.8	5
81.0	6
81.2	7
81.6	8
82.0	9
82.3	10
82.8	11
83.0	12
83.3	13
83.5	14
85.8	15
86.1	16
86.7	17

Graphs

Simple Distillation



Fractional Distillation



Questions

1. It is imperative for liquid to flow back down the fractionating column because this allows the liquid to condense into a more concentrated, purer, more volatile compound. As the vapour travels up the column, the cooler temperatures allow it to condense, trickle down, and then vaporize again. The repeated process not only purifies the liquid, but allows the column temperature to balance so that by the time vapour reaches the condenser, the vapour only consists of the pure, volatile compound.
2. The fractionating column should be kept at a constant temperature to ensure that the compound remains pure and volatile throughout its ascension in the column. If the temperature gradient is erratic, the compound will condense and re-vapourize inconsistently, and may not reach the condenser tube. Also, the thermometer may record a different temperature than the actual temperature of the boiling liquid, if the compound condenses before it reaches the thermometer.
3. $Pt = PaXa + PbXb$ However, at the boiling point of any liquid is the point at which its vapor pressure is equal to the pressure within the system (which in this case is equal to atmospheric pressure) therefore the vapor pressure of benzene at 81 degrees is 760 torr.
4. An increase in atmospheric pressure would mean the boiling temperature would increase. Looking at the ideal gas law $PV = nRT$, as the pressure increases, assuming that volume and number of moles remains constant, the temperature of the system or substance would also have to increase.
5. If the condenser has water flowing from the top to the bottom, less water flows through the condenser and therefore the tube is not fully immersed and not properly cooled. For the high heat capacity gas to condense, the condenser must be at its best possible cooling potential; with water flowing from the bottom to the top, ensuring maximum flow rate.
6. **Mole fraction A=A/total** =3/(3+1)=0.75
   Mole fraction B= 1-0.75=0.25
   $Pt = PaXa + PbXb$ (0.75)(350)=262.5 mm Hg (Pa) (0.25)(150)=37.5 mm Hg (Pb)
   Pt= 262.5+37.5= ***300 mm Hg***

Discussion

Distillation is a relatively quick way to separate different components of a solution. Distillation is based on the idea of a liquid's volatile properties, or the temperature at which it is evaporated. A solution of different compounds can have those compounds separated by gradually increasing the temperature of the solution, then condensing each component back into liquid via a cooled condenser tube.

Simple distillation is a streamlined version of distillation, in which the solution is boiled, the temperature recorded at the top of the distillation head, and then condensing the vapor in a cooled condenser tube, finally collecting the liquid in the receiving flask. Fractional distillation is a more accurate, but more time consuming method of distillation. The only difference between the two methods is the addition of the fractional column in fractional distillation. This column is

filled with an inert material, that increases the surface area, enriching the vapor and allowing better separation. As the vapor rises up the fractional column, it condenses, and flows back down, until it reaches high temperature areas and boils again. Repeating this enriches the vapor for a purer distillation at the cost of time.

As the data shows, we were unable to fully complete both distillations. Ideally, we should have boiled off all 25 mL of the solution, however due to time constraints we could only boil off most of the liquid. Given that we allowed the same amount of time for both distillations, we can see that since we only obtained 17 mL of solution in the fractional distillation that it took longer to distill.

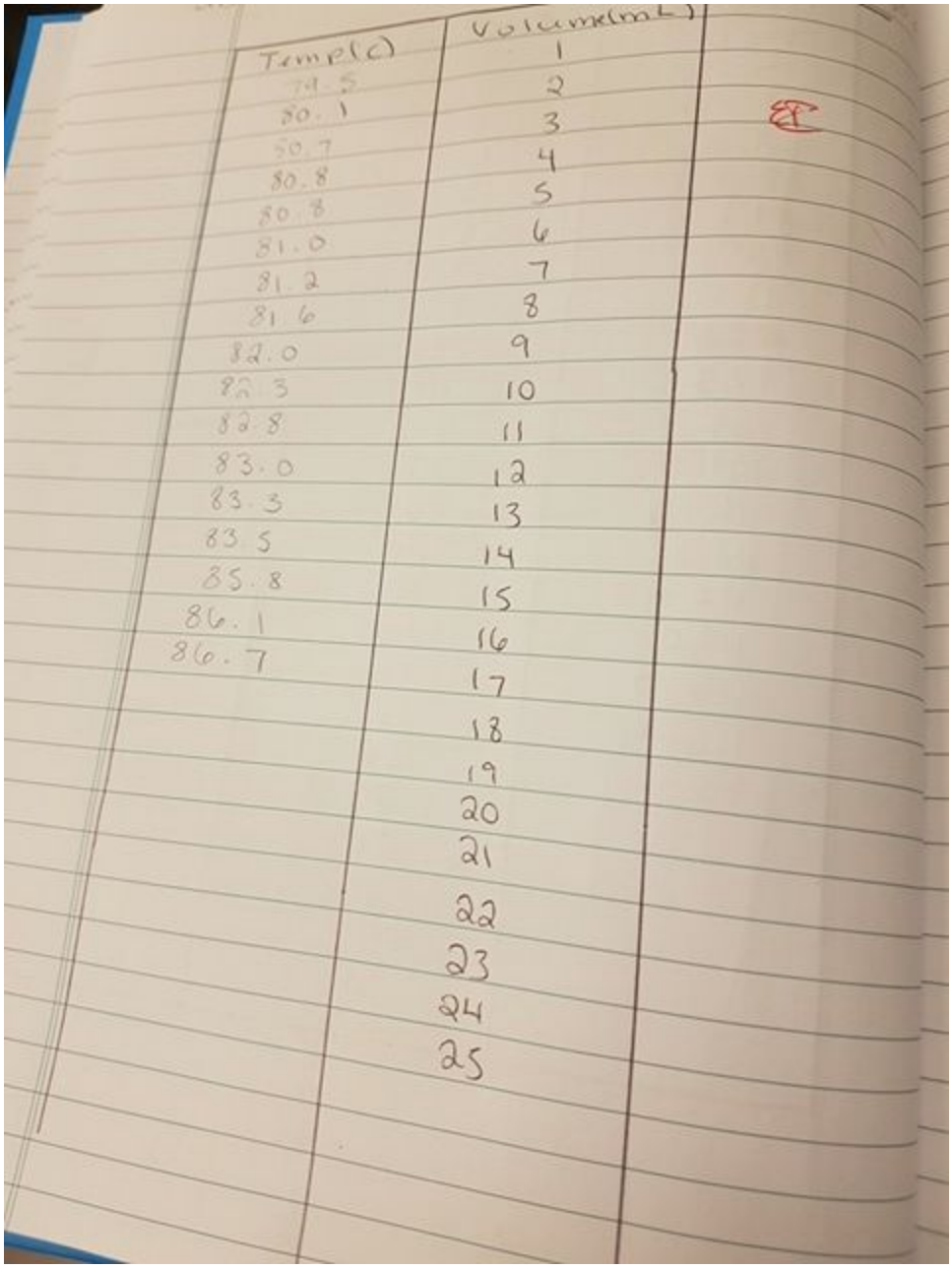
The data also shows the boiling point of the two compounds within the 50:50 solution; 2-propanol & 1-butanol. The more volatile liquid would have a lower boiling point and therefore would distill first, and ideally we would collect about 12.5 mL of this component; 2-propanol. We know that its boiling point must be around 81-82 degrees celsius, as we know that it was the more volatile liquid, and collection in the distillation flask began at about 81 degrees. Therefore, we can assume that collection of 1-butanol occurred after 50% (12.5 mL) of the solution has boiled off, which is supported by our simple distillation data. At about 11-12 mL, there was a large spike in temperature, indicating that the less volatile liquid had began to vaporize and reach the temperature. It was also around this time that a change in the solution of the receiving flask could be observed; similar to heat waves off of hot pavement, it was obvious that a new compound of different density was entering the receiving flask.

As previously mentioned, fractional distillation is a more accurate method of isolating components, however more time consuming. Our data shows that the temperature remained more constant throughout the experiment; therefore the process took longer, however the vapor and liquid in the receiving flask was purer than that of the simple distillation.

The most notable source of error was that we couldn't seem to fix the thermometer to the distillation head correctly. The end of the thermometer made constant contact with the glass tube, and therefore may have incorrectly measured the temperature of the gas. Additionally, our heating mantle was less effective, due to an apparent electrical problem and that it didn't nestle the distilling flask correctly, allowing much of the heat created to escape the system, and less heat to affect the solution inside the flask. Finally, there was a minor issue with the c-clamps used to hold the apparatus together. Due to the cramped space and angle of the apparatus, we had to sacrifice sturdiness and sealing of joints to allow other member of the lab to set up, and on occasion small amounts of vapor and very small amounts of liquid could be seen escaping the apparatus, particularly at the join between the distillation head and the condenser.

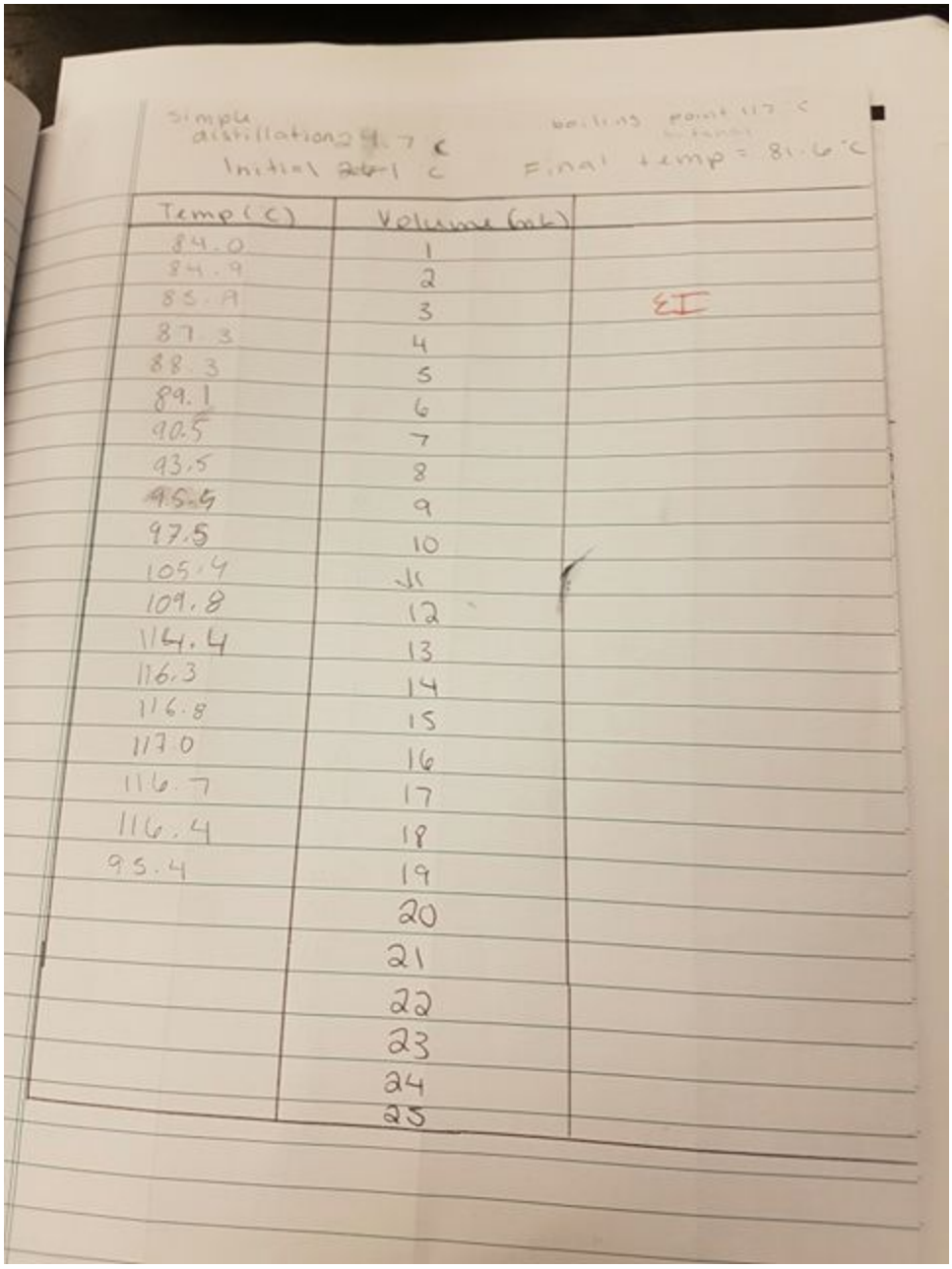
Raw Data

Fractional Distillation



Temp(C)	Volume(mL)
79.5	1
80.1	2
80.7	3
80.8	4
80.8	5
81.0	6
81.2	7
81.6	8
82.0	9
82.3	10
82.8	11
83.0	12
83.3	13
83.5	14
85.8	15
86.1	16
86.7	17
	18
	19
	20
	21
	22
	23
	24
	25

Simple Distillation



simple distillation 2

Initial [illegible] C

boiling point 117 C

Final temp = 81.6°C

Temp (C)	Volume (mL)	
84.0	1	
84.9	2	
85.9	3	EI
87.3	4	
88.3	5	
89.1	6	
90.5	7	
93.5	8	
95.4	9	
97.5	10	
105.4	11	
109.8	12	
114.4	13	
116.3	14	
116.8	15	
117.0	16	
116.7	17	
116.4	18	
95.4	19	
	20	
	21	
	22	
	23	
	24	
	25	