

Experiment 2: Purifying Chemicals by Distillation

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Procedure

As outlined in the lab manual (“Experiment 2: Purifying Chemicals by Distillation”, Organic Chemistry Laboratory Manual, Department of Chemistry, University of Ottawa, 2013, Exp. 2, pg. 6-7).

Tables and Graphs

Table 1: Simple distillation results

Volume (mL)	Temperature (°C)
1	43
2	50
3	52
4	54
5	55
6	56
7	57
8	58
9	59
10	60
11	62
12	64
13	65
14	67
15	68
16	69
17	70
18	71
19	72
20	74
21	74
22	74

Table 2: Fractional distillation results

Volume (mL)	Temperature (°C)
1	38
2	40
3	43
4	44
5	46
6	47
7	49
8	51
9	53
10	57
11	59
12	61
13	62
14	63
15	63
16	66
17	64
18	63
19	62
20	62

Figure 1: Temperature change per millimeter of distillate obtained during simple distillation

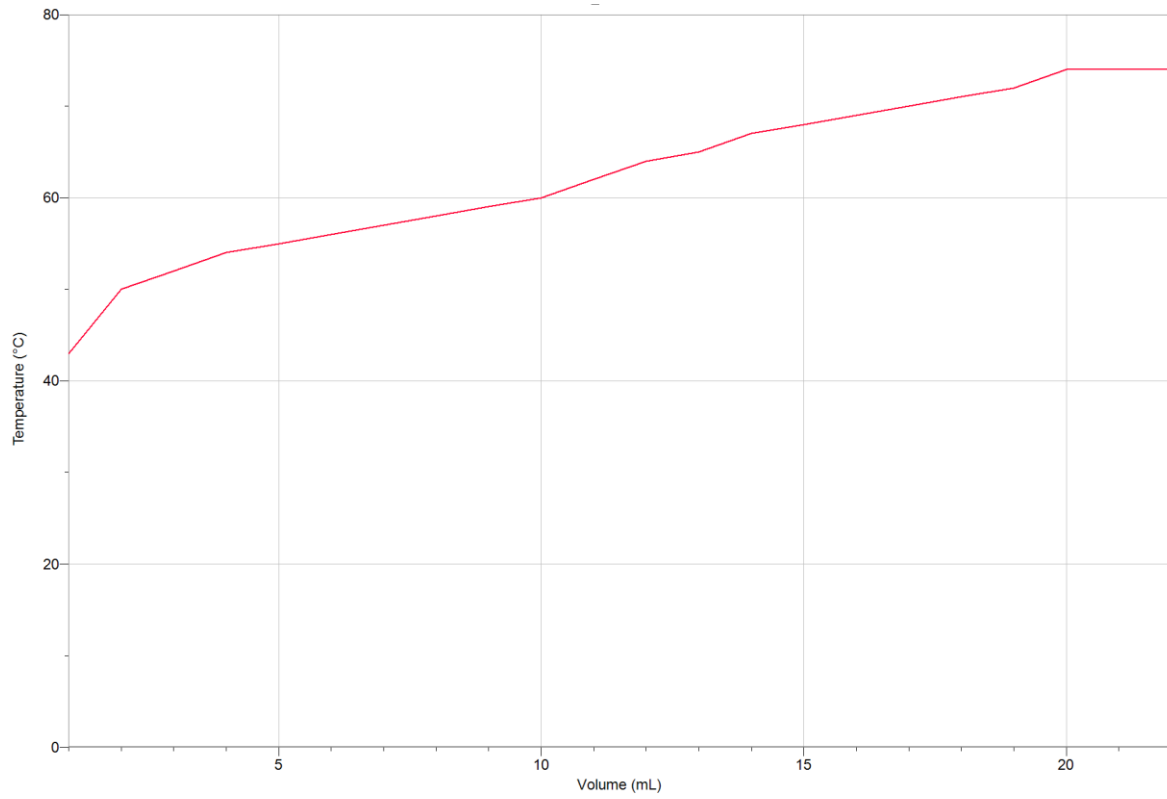
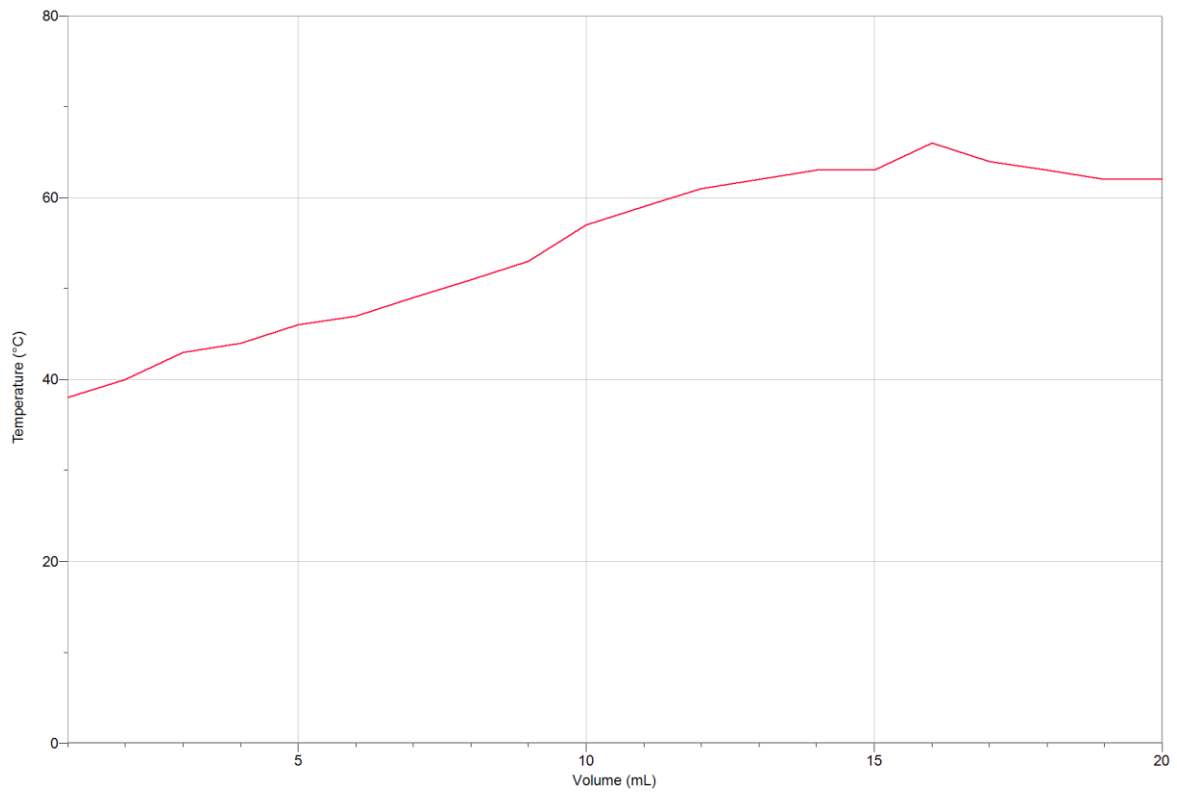


Figure 2: Temperature change per millimeter of distillate obtained during fractional distillation



Observations

Simple distillation

- the mixture is transparent & colourless solution
- as temperature increased, began to see condensation forming in the condenser
- condensed vapour slowly increased down the length of the condenser
- some of the condensed vapour began reforming into small liquid droplets sticking to the sides of the condenser
- despite watching this occur, it took a few minutes for the distillate to begin dripping down into the receiving flask
- by the end of the distillation, there was almost no liquid left in the distilling flask

Fractional distillation

- as condensed vapour slowly increased down the length of the condenser, some condensed vapours were also present near the top of the fractional column
- after the droplets from the condenser began dripping into the receiving flask, some of the previously condensed vapour at the top of the fractional column had also reformed into droplets and began dripping back into the distilling flask
- by the end of the distillation, a very small volume of liquid remained in the distilling flask

Discussion

- the mixture used in this experiment was a 50:50 mixture of dichloromethane:ethyl acetate
- the graph for simple distillation (Figure 1) displays a steadily increasing trend
- there is no distinct variation or shift in the trend to indicate when the distilling flask was enriched with one liquid or the other through effective removal of one liquid via distillation
- this indicates that the composition of the vapour did not slowly accumulate one of the liquids over time, but rather it accumulated a combination of both throughout, thus indicating a poor separation of the two compounds
- this information allows us to predict that there may be a large difference between the boiling points of dichloromethane and ethyl acetate ($\Delta T < 100\text{ }^{\circ}\text{C}$), thereby indicating that simple distillation is an ineffective method of purification
- the graph for fractional distillation (Figure 2) displays a similarly increasing trend, however the slope becomes negative after 16 mL
- this change in the trend of the graph indicates a shift towards the boiling point of one liquid from the other, since there would be a larger volume of one liquid in the distilling flask due to the removal of the other liquid via distillation
- the change in trend is very slight, however this is to be expected due to the information about the nature of the boiling points of the two liquids obtained in the simple distillation (i.e. that their boiling points are close)

- based on this information, we can predict that there was some separation of the two liquids; the degree of separation was very small, but still present
- although it was not a completely pure distillation, there was comparatively more separation than obtained in the simple distillation, which indicates a slight degree of success in distilling the mixture
- based on these results, we can predict that the more volatile liquid was most likely dichloromethane, as it is a nonpolar compound and is thus held together by weak van der Waals interactions and therefore requires less heat to evaporate (lower boiling point), whereas ethyl acetate is a polar compound (due to the presence of bond dipoles i.e. C-O) and thus requires more thermal energy to evaporate (higher boiling point) [1]

Sources of Error

- error may have arisen from using the same distillation flask (graduated cylinder) in the fractional distillation as in the simple distillation; the leftover distillate from performing the simple distillation may have contributed to the volume observed and recorded during the fractional distillation
- the graduated cylinder was clamped at a slight angle, which may have caused us to record inaccurate volumes; since the apparatus was arranged such that the graduated cylinder could not be situated on a flat surface, it was difficult to align it perfectly such that it was not at an angle
- the limitations of the lab equipment and provided space left a lot of room for human error in the recording of the temperature and volume; the temperature was consistently rising, thus only an approximation could be recorded; as well, relying on our eyes to approximate where the mercury was situated is not a reliable way to obtain data, thus leaving lots of room for error
- due to restraints in time, equipment, and space, we could only perform one trial of each distillation; a single trial provides a very limited amount of data and does not provide other data to which we can compare, therefore we do not know whether our data points contains significant outliers

Questions

1.
 - the boiling of toluene is 110.6 °C [3], whereas the boiling point of ethanol is 78.2 °C [4]
 - $\Delta T = 32.4 \text{ °C} < 100 \text{ °C}$
 - the difference in boiling points between toluene and ethanol is not large enough to successfully purify via simple distillation, however it may be possible to purify via fractional distillation, since ethanol would evaporate first due to its lower boiling point and could thus be collected in a receiving flask
 - this relies on whether the vapour pressure of ethanol is higher than that of toluene; if the corresponding properties of the two liquids satisfy Raoult's law [2], then a successful distillation could theoretically be ensured

2.
 - maintaining a uniform temperature gradient ensures that the only the compound with the higher boiling point will evaporate and reach the condenser, leaving the other compound to drip back down into the distilling flask
 - if the temperature increases too much, it may exceed the boiling point of the high-boiling point compound; the compound with the higher boiling point cannot return to the original flask, as it will only return to its liquid state upon coming into contact with the cool water in the condenser, resulting in an unsuccessful distillation
 - if the temperature is not high enough, it may not reach the boiling point of the low-boiling point compound; the compound with the lower boiling point will thus also drip back down into the receiving flask, resulting in an unsuccessful distillation
3.
 - at its boiling point, the vapor pressure of any liquid is equal to the pressure it was observed at [1], which is usually atmospheric pressure (1.0 atm, 760 mmHg, or 101.325 kPa)
 - therefore, the vapour pressure of dichloromethane at its boiling point is equal to whatever pressure it was observed at, most likely 1.0 atm
4.
 - since the boiling point is determined by the vapour pressure of a liquid and vapour pressure is equal to the pressure observed, increasing the atmospheric pressure therefore increases the boiling point of a liquid
5.
 - having cooling water enter the bottom of the condenser first allows for the water to effectively reach the entire volume of the condenser, allowing for maximized cooling of the hot vapours
 - water entering from the top would not fill the entire volume of the condenser, thus only cooling part of it and preventing effective cooling
6.
 - according to Raoult's law [2]:
 - $P_{\text{total}} = (P_{\text{A}^\circ})(N_{\text{A}}) + (P_{\text{B}^\circ})(N_{\text{B}})$
 - where P_{A° is the vapour pressure of compound A, P_{B° is the vapour pressure of compound B, N_{A} is the mole fraction of compound A in the mixture, and N_{B} is the mole fraction of compound B in the mixture
 - a 4:1 mixture indicates a mixture constituting 4/5 mol A and 1/5 mol B, therefore the mole fractions of A and B are 0.80 mol and 0.20 mol
 - based on the given information, we can conclude that $P_{\text{A}^\circ} = 350 \text{ mmHg}$, $P_{\text{B}^\circ} = 150 \text{ mmHg}$, $N_{\text{A}} = 0.80$, and $N_{\text{B}} = 0.20$
 - therefore:
 - $P_{\text{total}} = (P_{\text{A}^\circ})(N_{\text{A}}) + (P_{\text{B}^\circ})(N_{\text{B}})$
 - $P_{\text{total}} = (350 \text{ mmHg})(0.80) + (150 \text{ mmHg})(0.20)$
 - $P_{\text{total}} = 280 \text{ mmHg} + 30 \text{ mmHg}$
 - $P_{\text{total}} = 310 \text{ mmHg}$
 - the total vapour pressure of a 4:1 mixture of A and B is 310 mmHg

References

1. "Experiment 2: Purifying Chemicals by Distillation", Organic Chemistry Laboratory Manual, Department of Chemistry, University of Ottawa, 2013, Exp. 2, pg. 1
2. "Experiment 2: Purifying Chemicals by Distillation", Organic Chemistry Laboratory Manual, Department of Chemistry, University of Ottawa, 2013, Exp. 2, pg. 2, Fig. 3
3. National Center for Biotechnology Information. PubChem Database. Toluene, CID=1140, <https://pubchem.ncbi.nlm.nih.gov/compound/Toluene> (accessed on Feb. 6, 2020)
4. National Center for Biotechnology Information. PubChem Database. Ethanol, CID=702, <https://pubchem.ncbi.nlm.nih.gov/compound/Ethanol> (accessed on Feb. 6, 2020)

Raw Data

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Simple Distillation

mL	Temp (°C)	mL	Temp (°C)
1	43	22	74
2	50	23	—
3	52	24	—
4	54	25	—
5	55		
6	56		
7	57		
8	58		
9	59		
10	60		
11	62		
12	64		
13	65		
14	67		
15	68		
16	69		
17	70		
18	71		
19	72		
20	74		
21	74		

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Experiment 2

01/03/20

Fractional Distillation

ml	Temp (°C)	ml	Temp (°C)
1	38	22	—
2	40	23	—
3	43	24	—
4	44	25	—
5	46		
6	47		
7	49		
8	51		
9	53		
10	57		
11	59		
12	61		
13	62		
14	63		
15	63		
16	66		
17	64		
18	63		
19	62		
20	62		
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