Purifying by Distillation

Experiment 2

Written by:

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CHM1321 Z05

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Procedure

As described in the lab manual. (Durst et al., *Organic Chemistry Laboratory Manual,* exp. 2, 2019)

Data

Simple Distillation: Experiment #1

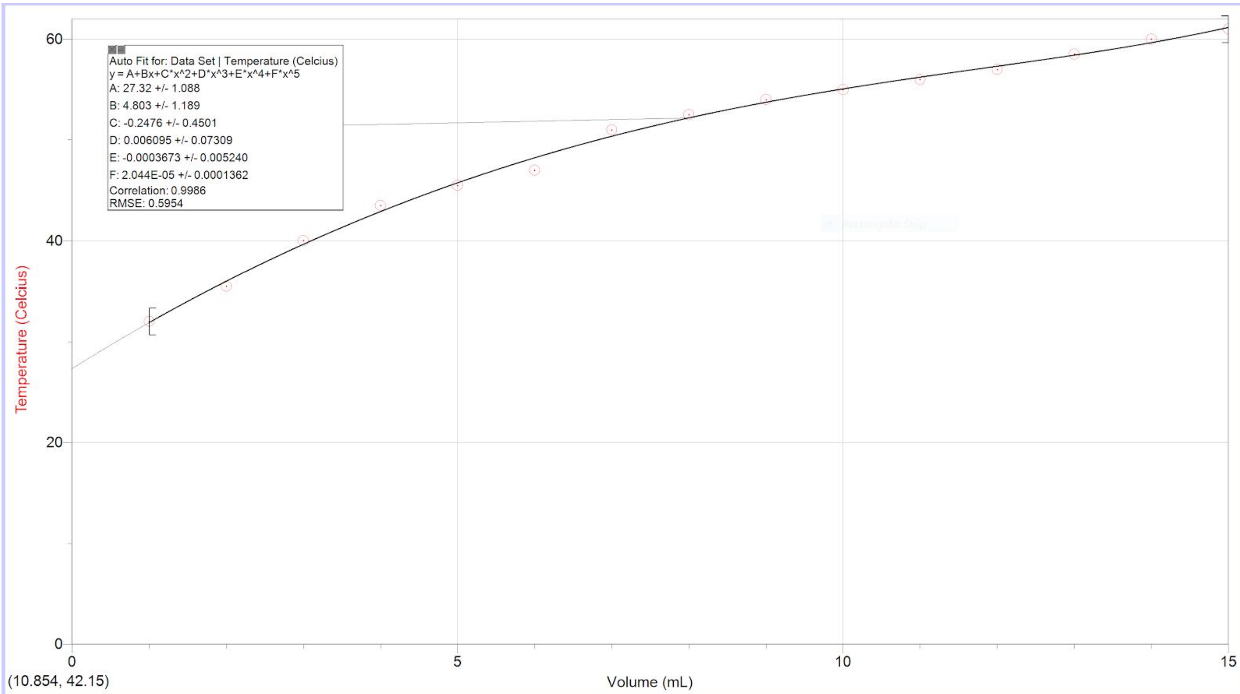
Volume (mL)	Temperature (℃)
0	27.0
1	44.5
2	49.0
3	51.0
4	52.0
5	53.0
6	54.5
7	55.5
8	56.5
9	57.5
10	59.0
11	60.5
12	62.0
13	63.5
14	64.5
15	66.0

Fractional Distillation: Experiment #2

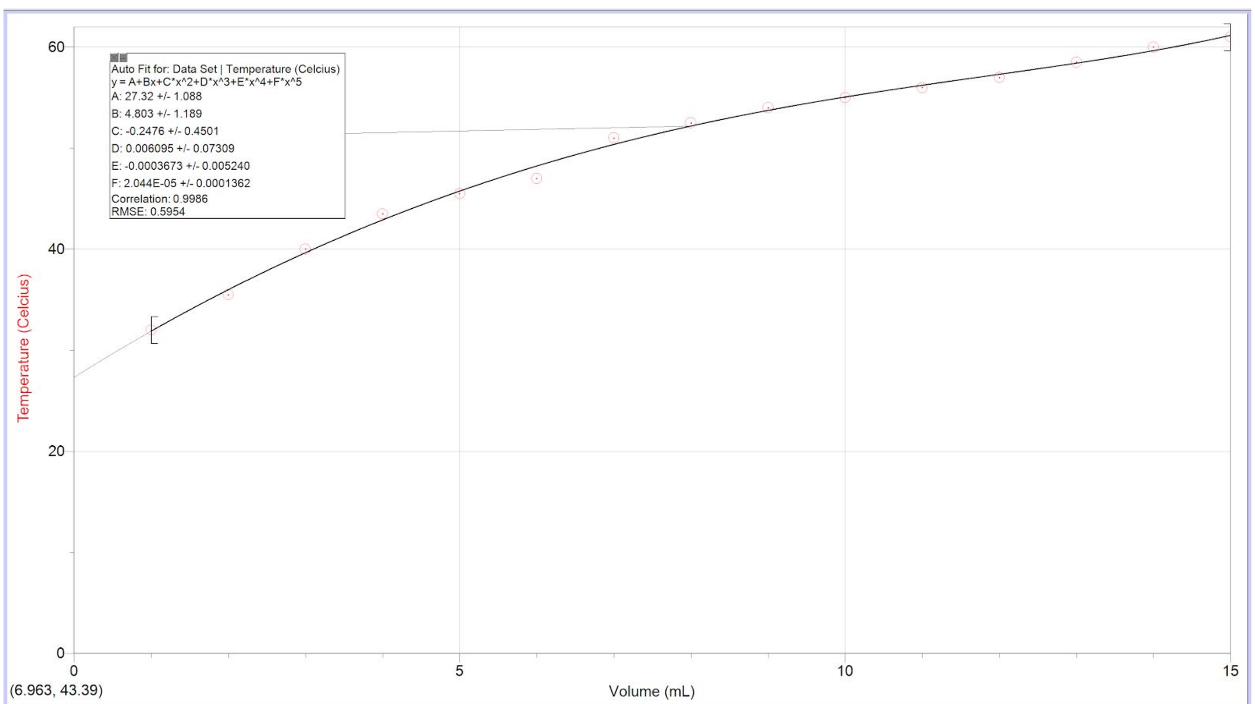
Volume (mL)	Temperature (℃)
0	27.0
1	32.0
2	35.5
3	40.0
4	43.5
5	45.5
6	47.0
7	51.0
8	52.5
9	54.0
10	55.0
11	56.0
12	57.0
13	58.5
14	60.0
15	61.0

Graphs

Simple Distillation (Experiment 1):



Fractional Distillation (Experiment 2):



Questions

1. Is it possible to separate a mixture of ethanol and toluene via distillation? Explain.
   Yes it is possible to separate a mixture of ethanol and toluene using distillation. Ethanol has a boiling point of 78.3℃ and toluene has a boiling point of 110.6℃. Distillations work by boiling off one or more compounds to get to a pure and more desired substance. Ethanol and toluene have a difference of boiling points of 32.3℃. This difference of temperature allows the two substances to be able to be seperated during distillation. Water, which has a boiling point of 100℃, and ethanol are able to be separated via distillation. Ethanol and water's difference in boiling points is only 21.7 ℃, which means that toluene and ethanol can be separated as their difference in boiling points is higher.

2. Fractionating columns normally work better if they are insulated in order to maintain a smooth temperature gradient in the column. Why is it important to maintain a uniform temperature gradient in a fractionating column?
   It is important to maintain a uniform temperature gradient in the fractionating column because if the temperature is too high, the condensing liquid in the column will not be able to flow back down to the distilling flask because of how much vapour will be moving up the column. This results in a flood, which is a pocket of liquid forming in the column. If a flood occurs, the efficiency of the distillation is greatly decreased as the distillation is essentially restarted at pocket of liquid.

3. The boiling point of dichloromethane is 39.6℃. What is the vapour pressure of dichloromethane at this temperature?
   The vapour pressure of dichloromethane at 39.6℃ is equal to the atmospheric pressure (not given) at the boiling point.

4. What effect does an increase in atmospheric pressure have on the boiling point of a liquid?
   When the vapour pressure of a liquid matches the atmospheric pressure the boiling point is reached. Increasing the atmospheric pressure would in turn increase the boiling point of a liquid, and decreasing the atmospheric pressure would in turn decrease the boiling point. Reducing the atmospheric pressure lessens the pressure pushing the liquid molecules down, so they can more easily transition into the gas phase.

5. Why is it important to have cooling water enter the bottom of the condenser and not at the top.
   Cold water enters the bottom of the condenser so that the condenser will be completely full of water and will do its job to condense the vapour. When the system has been setup

properly you cannot see the water flowing through, and it will look as if there is no water in the condenser at all.

6. Compound A has a vapour pressure of 350 mm Hg at 95℃ whereas compound B has a vapour pressure of 140 mm Hg at the same temperature. If A and B are miscible, what is the vapour pressure of a 4:1 mixture of A and B at 95℃.
   Using Raoult's Law, the vapour pressure of a 4:1 mixture of A and B at 95℃ is 308 mm Hg. There are a total of 5 moles in the solution, meaning the mole fractions are ⅘ for A and ⅕ for B. Determined using the following calculations:
   $P_A = \frac{4}{5} \cdot 350\ mm\ Hg\ = 280\ mm\ Hg$
   $P_B = \frac{1}{5} \cdot 140\ mm\ Hg\ = 28\ mm\ Hg$
   $P_{total} = 280\ mm\ Hg\ +\ 28\ mm\ Hg\ =\ 308\ mmHg$

Observations (4-6)

- The fractional distillation took longer then the simple distillation (took longer to heat up and also get 15 mL of the solution in volumetric flask)
- All solutions were colourless and transparent before and after the distillations
- More condensation around the part of the condenser closes to the solution where it heats up
- Solution accumulated in volumetric flask had bubbles in it
-

Discussion

Neither of the distillations observed in this experiment were very efficient. In an effective simple distillation, the Temperature vs Volume graph has a more dramatic and noticeable slope increase as volume increases. This would be because the more volatile solution in the mixture would vapourize first, which would be the lower portion of the graph. As volume continues to increase, temperature should begin to rise a little quicker, this would be as the second part of the mixture begins to vapourize. Then, towards the end of the graph, the temperature would begin to level out, this will be when there is no more of the less volatile solution left. While the trends at the beginning and the end of the graph of the simple distillation match what was expected, the middle portion did not. Instead, the temperature increased gradually, which does not reflect an effective distillation.

In an effective fractional distillation, the same beginning and end trends are seen as in an effective simple distillation. However, in the middle portion of the graph, there would be a drastic temperature increase as the less volatile solution is almost completely vapourized, and has volume increases the graph levels out much more quickly. For this distillation, none of these trends were observed. The temperature increased at a constant rate and had no huge increase in temperature. The slope of the graph increases gradually as it did during the simple distillation, meaning again, this distillation was not effective.

The equipment required for a simple distillation is: a distilling flask, a graduated cylinder as the receiving flask, three clamps, distillation head, thermometer, thermometer adapter, condenser, vacuum takeoff adapter, 2 hoses to connect to the condenser, a stir plate, and an elevation flask.

Equipment required for a fractional distillation is the same as for simple distillation except it also requires a fractionating column.

This technique proved to be somewhat difficult. The setup has to be precise, as does the temperature of the heating mantle. If the heating mantle was turned up too high, the contents might have vapourized too quickly and would have been more difficult to measure the temperature changes as they would have occurred too quickly. Besides the heat and setup specificity, the rest of the technique was relatively easy. After the contents began to boil, the temperature was measured per milliliter of recondensed product collected, and the experiment for the fractional distillation remained the same except for adding the fractionating column to the apparatus.

During the simple distillation, the product will almost never be purely one reactant or the other as simple distillations are not as effective. At the very beginning the product may be completely composed of the reagent with the lower boiling point, but after the second reagent begins to boil the product is never pure. During an effective fractional distillation, the beginning portion of the collected products will be purely the lower boiling point reagent for a longer amount of time, as during a fractional distillation the lower boiling point reagent boils out almost completely before the higher boiling point reagent boils and appears in the products. Since the fractional distillation done was not effective, as shown on the graph, the products are not pure compared to what they should be at that point.

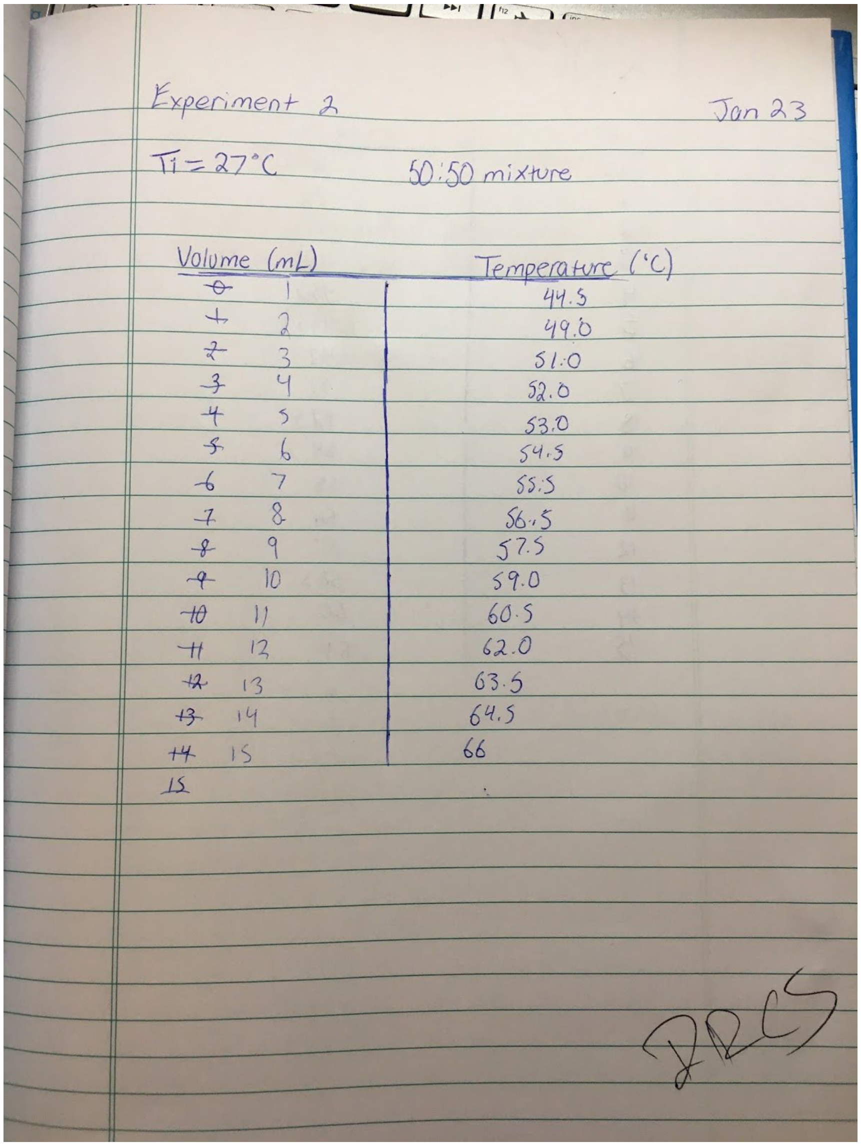
During the fractional distillation, the nature of the starting products will go from the 50:50 mixture, to being mostly if not entirely the higher boiling point reagent, as the lower one has been boiled out. Similarly in the simple distillation, it will go from the 50:50 mixture to being mostly comprised of the less volatile reagent, but there will still be some remnants of the more volatile reagent as simple distillations are not as effective as a fractional distillation.

Conclusion

It was predicted that both the simple and fractional distillation would display a rapid and noticeable temperature change, distinguishing between the evaporation of each reagent in the 50:50 mix. However, the results were inconclusive as neither of the graph's display these predictions, meaning there was no way to describe what the different boiling points of the two reagents were. The fractional distillation was predicted to have a more noticeable difference in the change of temperature to show the two different evaporation, but the results did not show this as there was no rapid increase of temperature.

Raw Data

Hope's Raw Data



Experiment 2 Jan 23

Ti = 27°C 50:50 mixture

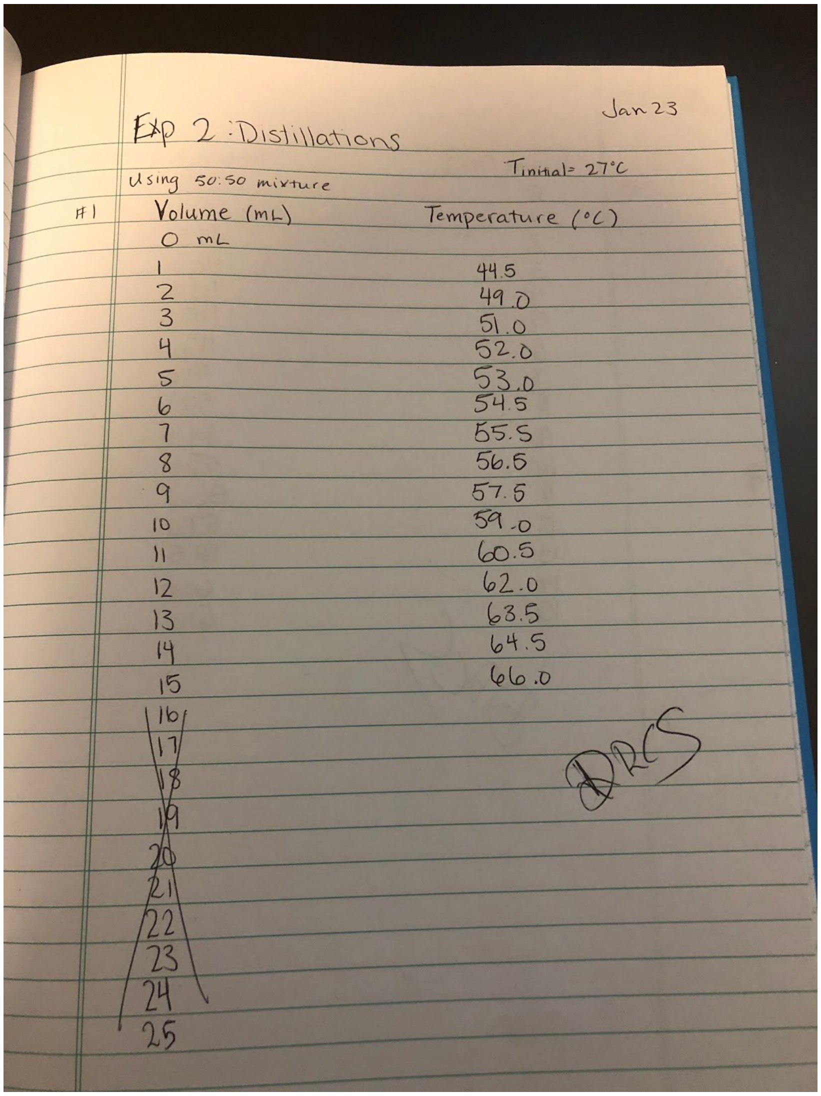
Volume (mL)		Temperature (°C)
~~0~~	1	44.5
~~1~~	2	49.0
~~2~~	3	51.0
~~3~~	4	52.0
~~4~~	5	53.0
~~5~~	6	54.5
~~6~~	7	55.5
~~7~~	8	56.5
~~8~~	9	57.5
~~9~~	10	59.0
~~10~~	11	60.5
~~11~~	12	62.0
~~12~~	13	63.5
~~13~~	14	64.5
~~14~~	15	66
~~15~~		

DRCS

DRCS

V	T
0	27°C
1	32
2	35.5
3	40
4	43.5
5	45.5
6	47
7	51
8	52.5
9	54
10	55
11	56
12	57
13	58.5
14	60
15	61

Hannah's Raw Data



Exp 2: Distillations — Jan 23

Using 50:50 mixture — $T_{initial} = 27°C$

#1

Volume (mL)	Temperature (°C)
0 mL	
1	44.5
2	49.0
3	51.0
4	52.0
5	53.0
6	54.5
7	55.5
8	56.5
9	57.5
10	59.0
11	60.5
12	62.0
13	63.5
14	64.5
15	66.0
~~16~~	
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~~23~~	
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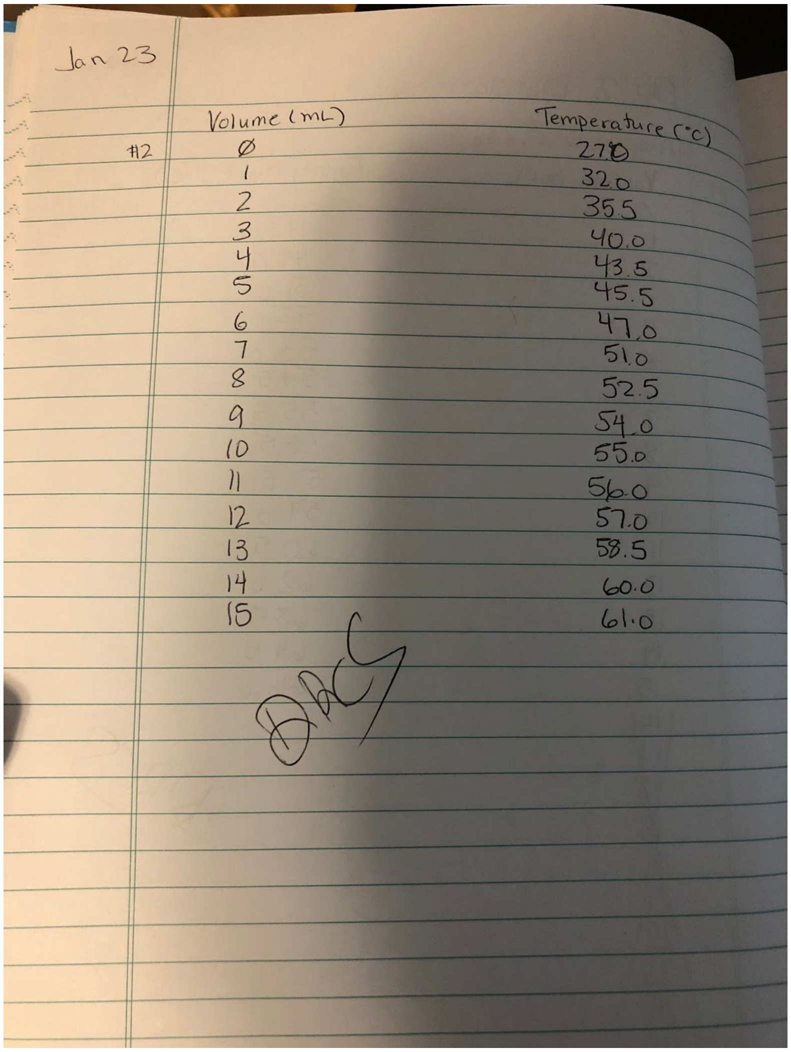

Jan 23

#2

Volume (mL)	Temperature (°C)
Ø	27.0
1	32.0
2	35.5
3	40.0
4	43.5
5	45.5
6	47.0
7	51.0
8	52.5
9	54.0
10	55.0
11	56.0
12	57.0
13	58.5
14	60.0
15	61.0

<u>References</u>

Durst et al., *Organic Chemistry Laboratory Manual,* exp. 2, 2019

COURSE: CHM 1321 **TA Name:** Daliare Regis
YOUR NAME (PRINT): Hope Boyle **SIGNATURE:** Hope Boyle

CONFIDENTIAL PEER EVALUATION FORM FOR EXPERIMENT 2

Each team member must submit one assessment form evaluating each other member of the team.
Teams will consist of 2 (max 3) members for reports.
You may edit this form.
Do not share or discuss the contents or possible contents of this assessment with others.

In assessing the work of your fellow team members, consider the following aspects:

- Quality of work
- Contribution to the work as a whole
- Ability to get along with others
- Improvements when asked to correct

Team member name	Comments	Grade (/5)
Hannah Rr Prozak	Great partner	5

A – Excellent (5) **B: Great (4)** **C: Good (3)** **D: Fair(2)** **F: Poor (1)**

Note: Do not evaluate yourself on this form