Purifying Chemicals by Distillation

Laboratory #2

DATE:Tuesday February 5, 2019

University of Ottawa

Organic chemistry (CHM1321)

Introduction:
The goal of this laboratory is to take a 50:50 concentration mixture and purify it by simple as well as fractional chemical distillation, and observe the process. Distillation purification can take place by heating a solution. In result of heating the pure liquid solution evaporates into a separate container and leaves behind the unwanted solid particles. Although distillation purification can also take place by heating a mixture consisting of two or more liquids, as performed in this laboratory.The mixture consists of two different solutions at a 50:50 ratio, and as these are two different solutions they have different temperatures at which they become gasses. In result one hypothesizes that there will be a temperature change of the solution, when the solution is halfway distilled, since one of the solutions in the 50:50 mixture will has evaporated, and the other will not yet have reached the activation energy (received by temperature) needed to evaporate since this liquid has a larger molar heat capacity. Therefore, the temperature will go down then rise again as the second solution gains more energy and its temperature increases.

Lichtarowicz, M. (2019). Distillation. Retrieved from <http://www.essentialchemicalindustry.org/processes/distillation.html>

Libretexts. (2018, April 02). 7.21: Simple Distillation. Retrieved from https://chem.libretexts.org/Bookshelves/Organic_Chemistry/Book:_Organic_Chemistry_Lab_Techniques_(Nichols)/7:_Technique_Summaries/7.21:_Simple_Distillation

Procedure:
Please refer to Laboratory manual)Experiment 2: Purifying Chemicals by Distillation
Section)The Experiment: Experiment 1 and 2 pages 7,8.
Reference)Department of Chemistry, University of Ottawa, 10 Marie Curie Priv, Ottawa, ON, K1N6N5
September 2013

Materials:
- 50:50 dichloromethane; Ethyl Acetate
- Thermometer
- Distillation head
- Extension clamp
- Distillation flask
- 2 Clips
- Thermometer adapter
- Vacuum takeoff adapter
- Condenser
- 2 Clamps
- Receiving flask
- Fractionating column

Tables of distillations:

Volume(ml)	Temperature Simple Distillation (°C)	Temperature Fractional Distillation(°C)
1	46	42
2	46	42
3	48	44
4	49	45
5	50	45
6	50	46
7	51	47
8	53	48
9	54	50
10	55	51
11	56	53
12	57	56
13	59	61
14	60	65
15	62	67

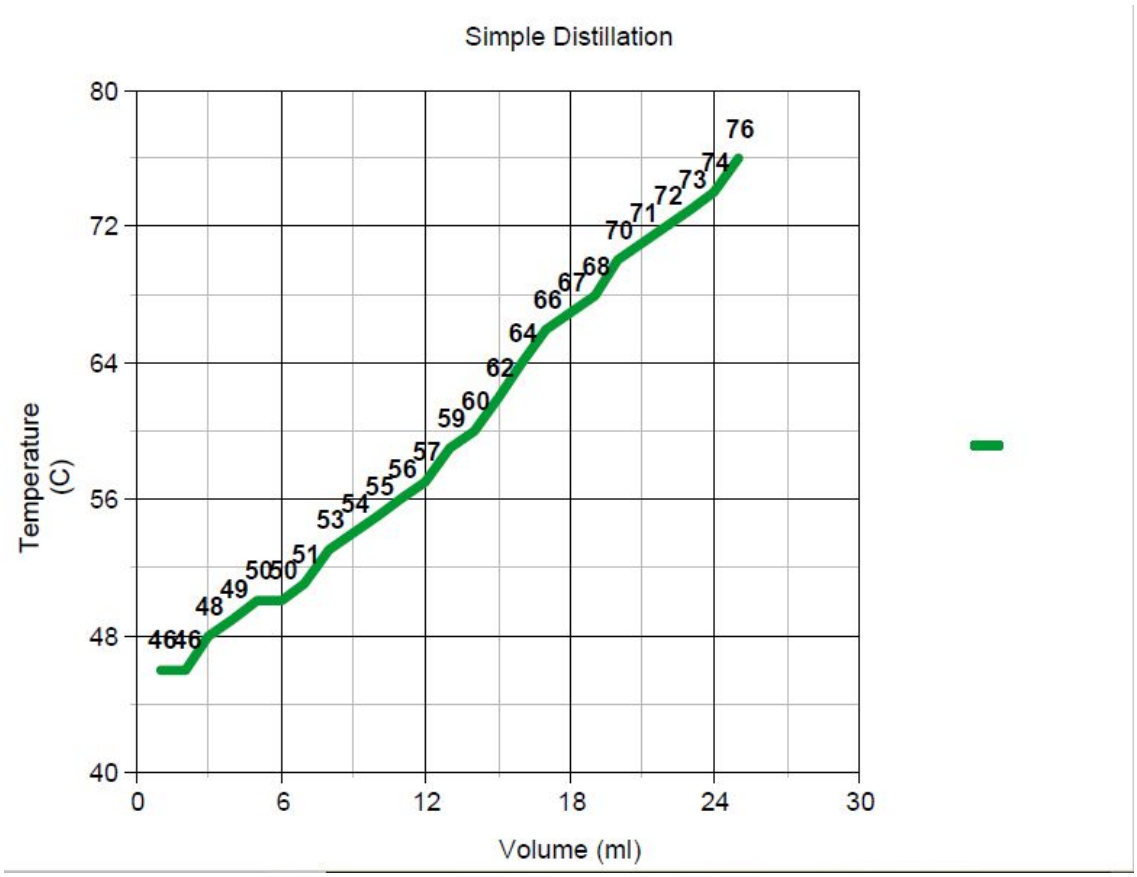
16	64	70
17	66	72
18	67	72
19	68	73
20	70	73
21	71	74
22	72	74
23	73	75
24	74	75
25	76	76

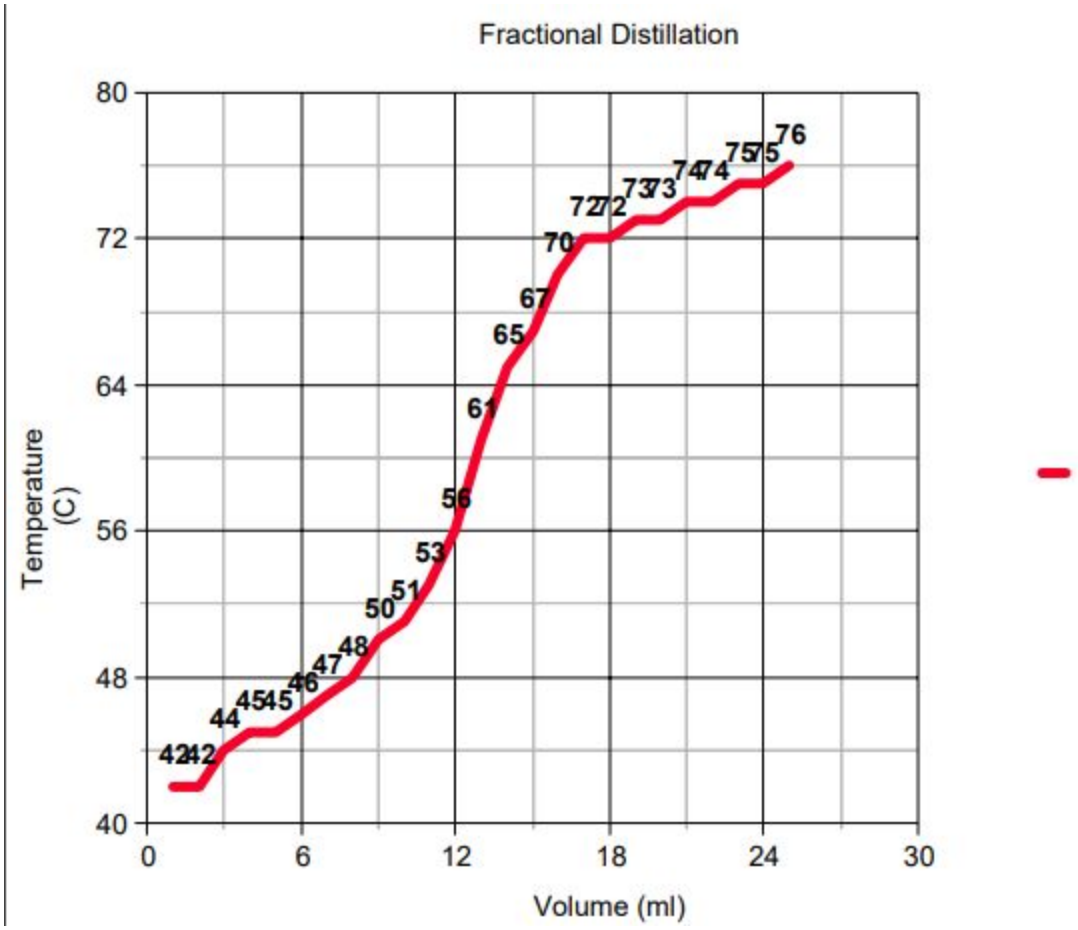
Observations:

- Simple distillation
    - When one observes this process one can see that during the first 13 ml of distillation the temperature rises at a faster pace. After that the temperature continues to rises, although at a slower and even rate.
    - It has a higher starting point in terms of temperature because the vapour is more a mixture, rather than just the low boiling point compound.

- Fractional Distillation
    - Temperature rises during the 1st 13ml fast. Then it levels off towards the end.
    - Has a lower starting point in temperature due to the vapour being mainly made of the lower boiling point compound.

Graph:


Simple Distillation
Temperature (C)
Volume (ml)
80
72
64
56
48
40
0
6
12
18
24
30
46
46
48
49
50
50
51
53
54
55
56
57
59
60
62
64
66
67
68
70
71
72
73
74
76



Questions:

`1. Yes, it is possible. However, there is no temperature difference between the two of 100 C or more. Thus, simple distillation would not create a pure extraction. A percentage of toluene would vapourize with the ethanol you are trying to extract (toluene has the higher boiling point). Fractional distillation is more preferred, as the vapourized toluene would re-condense onto the fractionating column and back into the initial flask. The larger the surface area of the fractionating column the better the separation between the compounds will be.

2. You maintain a uniform temperature in the fractionating column because if there is too much heat in the fractionating column the recondensed liquid would vapourize of the vapour heat. There will be no liquid flow, which results in a pocket and flooding. Thus, you would have to start again.

3. The boiling point of a compound is when the vapour pressure is equal to the atmospheric pressure. The standard atmospheric pressure is 1.04atm.

4. You would need more heat to reach boiling point if the atmospheric pressure was higher. The boiling point is when vapour pressure equals atmospheric pressure. In order to reach a higher vapour pressure, you need to add more heat.

5. The water must enter from the bottom so that it reaches all over the tube. If it came from the top, then there would only be a skinny stream, thus only a portion of the vapour would get condensed.

6. $P_{Total} = 1/5P_B + 4/5P_A$
= ⅕(140 mm Hg) + ⅘ (350 mm Hg)
= 308 mm Hg.
Thus, the vapour pressure of the mixture are 95 C is 308 mm Hg.

Discussion:

- The simple distillation graph shows a more linear relationship. This is correct. The temperature required increases at a steady pace because the composition of the vapour is a mixture. The vapour contains more of lower boiling point compound than the one with the higher boiling point. It is running out of the lower boiling point compound faster than the other. Hence the steady linear increase in temperature. Overall, this is all due to simple distillation not being as precise in mixtures sharing close boiling points.
- The fractional distillation graph is more exponential. There is a sudden increase in temperature, before it begins to level off again. This is correct. In this distillation the vapour being extracted is less of a mixture due to the fractionating column condensing the lower boiling point compound back into the flask. Therefore, the graph acts like a pure compounds almost, meaning the temperature stays constant. Once the compound with the boiling point runs out, there is only the compound with the higher boiling point left. Thus the temperature increases drastically in order to vapourize what was left behind.
- Overall, the data tells you that there is not a good separation in the simple distillation. The slowly curved/linear graph indicates that the composition of the vapour is always a mixture.
- The fractionating distillation graph has a better separation. The quick spike up in temperature indicates that the separate compounds, with different boiling points, from the mixture were being vaporized individually.
- We use distillation in many forms in the real world. For instance, we heat up salt water, in order to separate the water from the salt, thus making fresh water we can drink.

Error:

- Some of the equipment was not clean. This equipment was in contact with the solution used, thus could have affected the speed in which the compound's vapourized.

- Some solution spilled thus we lost 2 ml of our data towards the end, and has to make an educated estimate. Next time we will make sure we handle the solutions more carefully.
- Slight error when trying to see with the naked eye if the solution had reached the marks of volume on the graduated cylinder, in order to record our results.

Conclusion:
Overall, the 50:50 Dichloromethane: Ethyl Acetate could be distilled. However, it was better distilled using the fractionating method rather than the simple distillation method.