CHM1321 Lab 2 - Experiment 2 lab report for for Sandro Gambarotta's class.

Organic Chemistry I (University of Ottawa)

Purifying Chemicals by Distillation (Experiment 2)

Author:

CHM1321 Section B6

Demonstrator:

Lab Partner:

February 1st, 2018

Department of Chemistry

University of Ottawa

Protocol:

Please refer to the lab manual titled “Experiment 2: Purifying Chemicals by Distillation” by Dr. Tony Durst et al. on pages 25-27.

- Aluminum foil was placed onto the apparatus (mostly around the distilling flask and distillation head) in order to provide further insulation.

Observations:

- The 50:50 mixture of 2-propanol and 1-butanol was a clear and colourless liquid. There was a strong odour coming from this mixture.
- It took a several minutes for the temperature to begin rising for both the simple and fraction distillation, but continued to rise more quickly afterwards.
- The simple distillation seemed to just continuously increase in temperature while the fractional distillation increased to roughly 70˚C, cooled down, then rose once again.
- The condenser became quite cold for both the simple distillation as the distillation came to a completion.
- The general tendency was a higher temperature equated to a higher drip rate into the receiving flask until nearing the end of distillation. The last measurement (25 mL), was so extremely slow that it was skipped for both distillations.

Data Tables:

Table 1 – Simple distillation volume in receiving flask at given temperature

Volume (mL)	Temperature (˚C)
0	21.3
1	-*
2	-*
3	-*
4	55.0
5	58.3
6	61.9
7	66.0
8	68.2
9	69.4
10	71.5
11	71.8
12	72.1
13	72.9
14	73.1
15	73.2
16	73.3
17	74.7
18	74.9
19	78.8
20	82.1
21	78.2
22	98.9
23	90.6
24	68.7

*These temperature values were not recorded due to error on thermometer reading

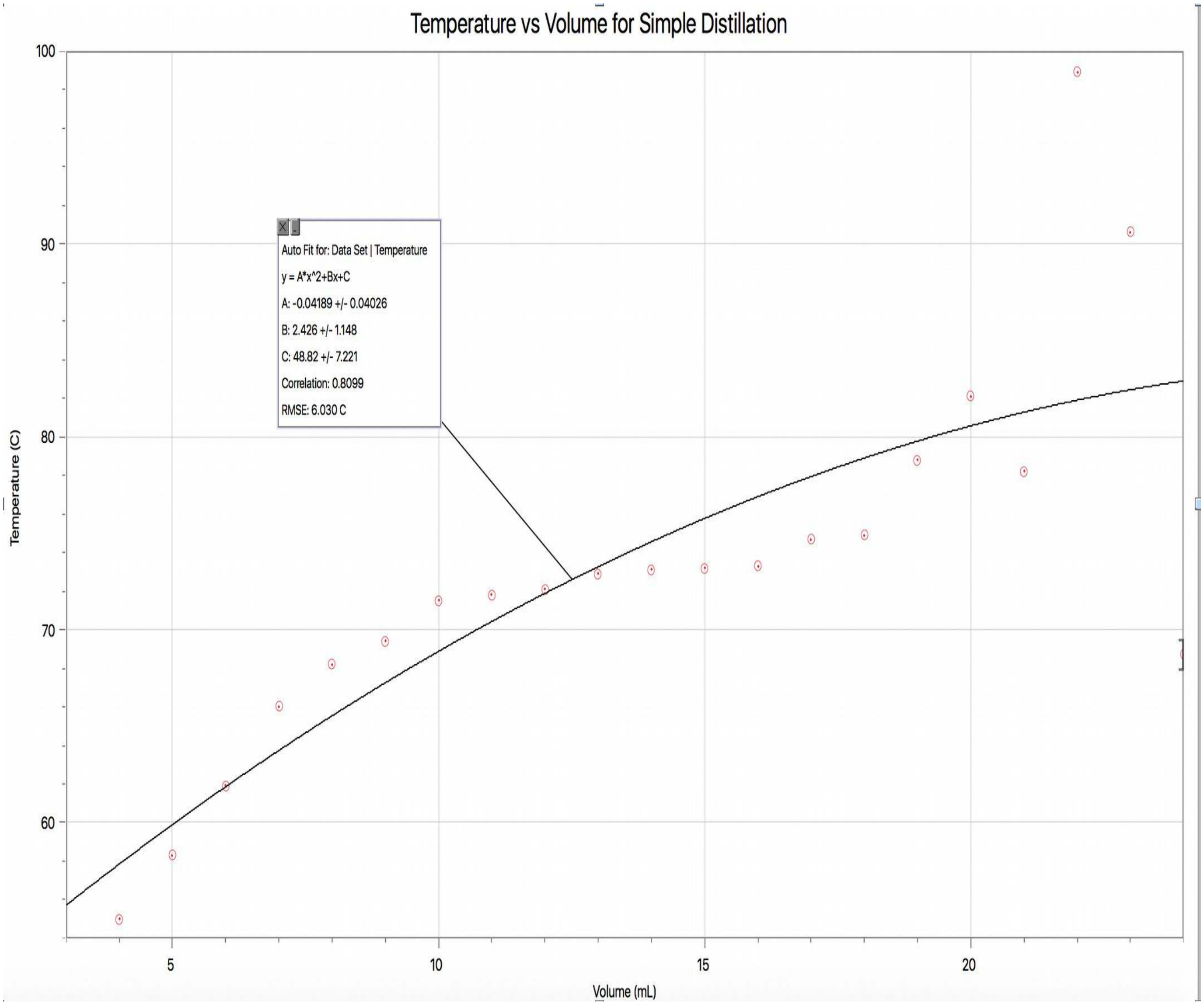
Table 2 – Fractional distillation volume in receiving flask at given temperature

Volume (mL)	Temperature (˚C)
0	24.8
1	55.0
2	61.3
3	65.6
4	68.5
5	70.4
6	70.5
7	70.8
8	68.5
9	68.6
10	66.2
11	65.0
12	62.3
13	60.2
14	60.0
15	60.2
16	63.9
17	69.3
18	73.4
19	75.9
20	76.4
21	76.3
22	74.1
23	70.7
24	54.1

Table 3 – Fractional distillation volume in receiving flask at given temperature borrowed from

Volume (mL)	Temperature (˚C)
2	81
3	81.6
4	81.8
5	82.1
6	82.4
7	82.8
8	83.3
9	83.8
10	84.4
11	85.5
12	87.3
13	89.1
14	95.1
15	106.9
16	114.2
17	115.6
18	115.7
19	115.7
20	115.7
21	115.7
22	115.7
23	115.6

Graphs of Distillations:



```
Auto Fit for: Data Set | Temperature
y = A*x^2+Bx+C
A: -0.04189 +/- 0.04026
B: 2.426 +/- 1.148
C: 48.82 +/- 7.221
Correlation: 0.8099
RMSE: 6.030 C
```

Figure 1 – Temperature (˚C) vs Volume (mL) collected in receiving flask in simple distillation with quadratic curve of best fit

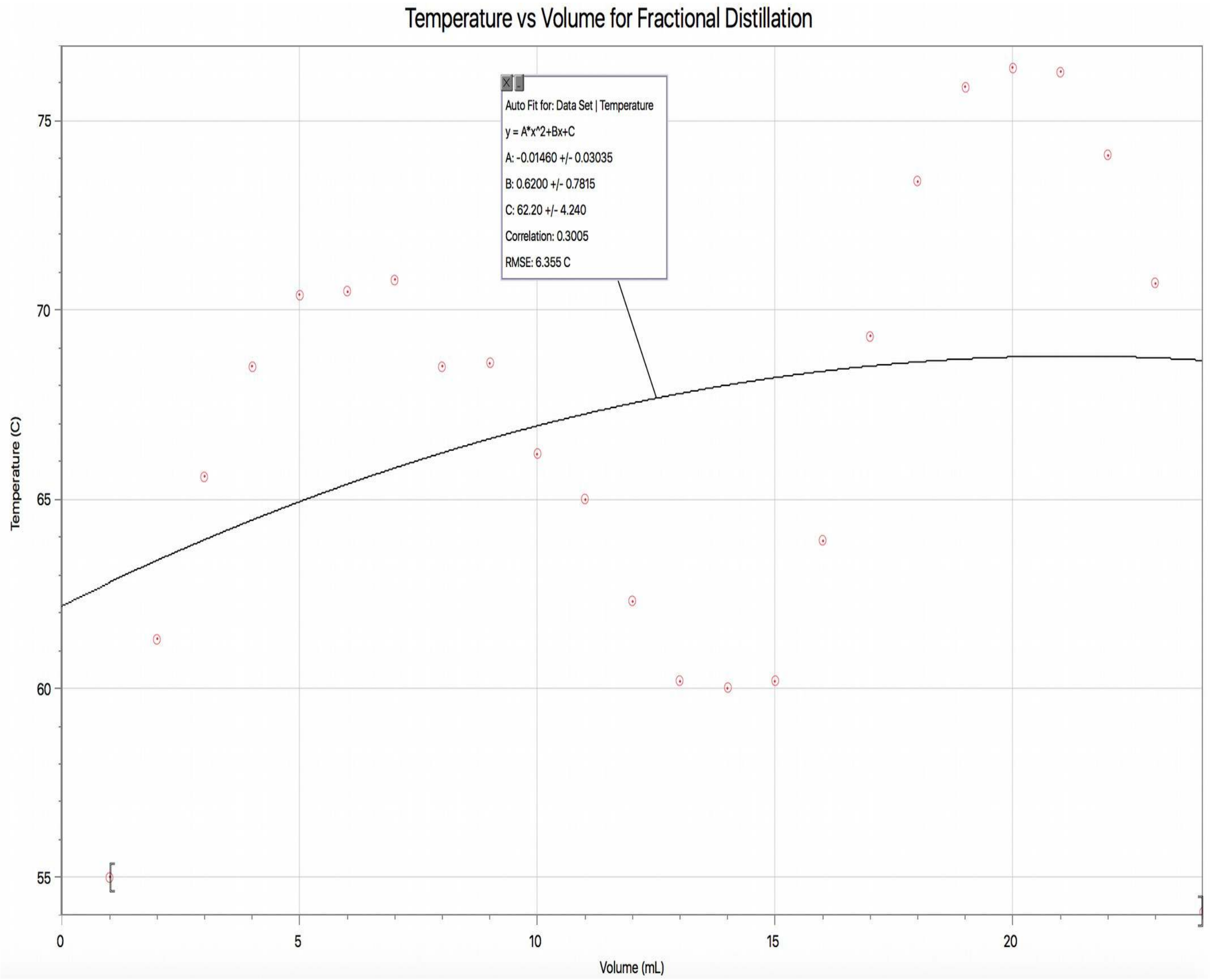


Figure 2 – Temperature (˚C) vs Volume (mL) collected in receiving flask in fractional distillation with quadratic curve of best fit

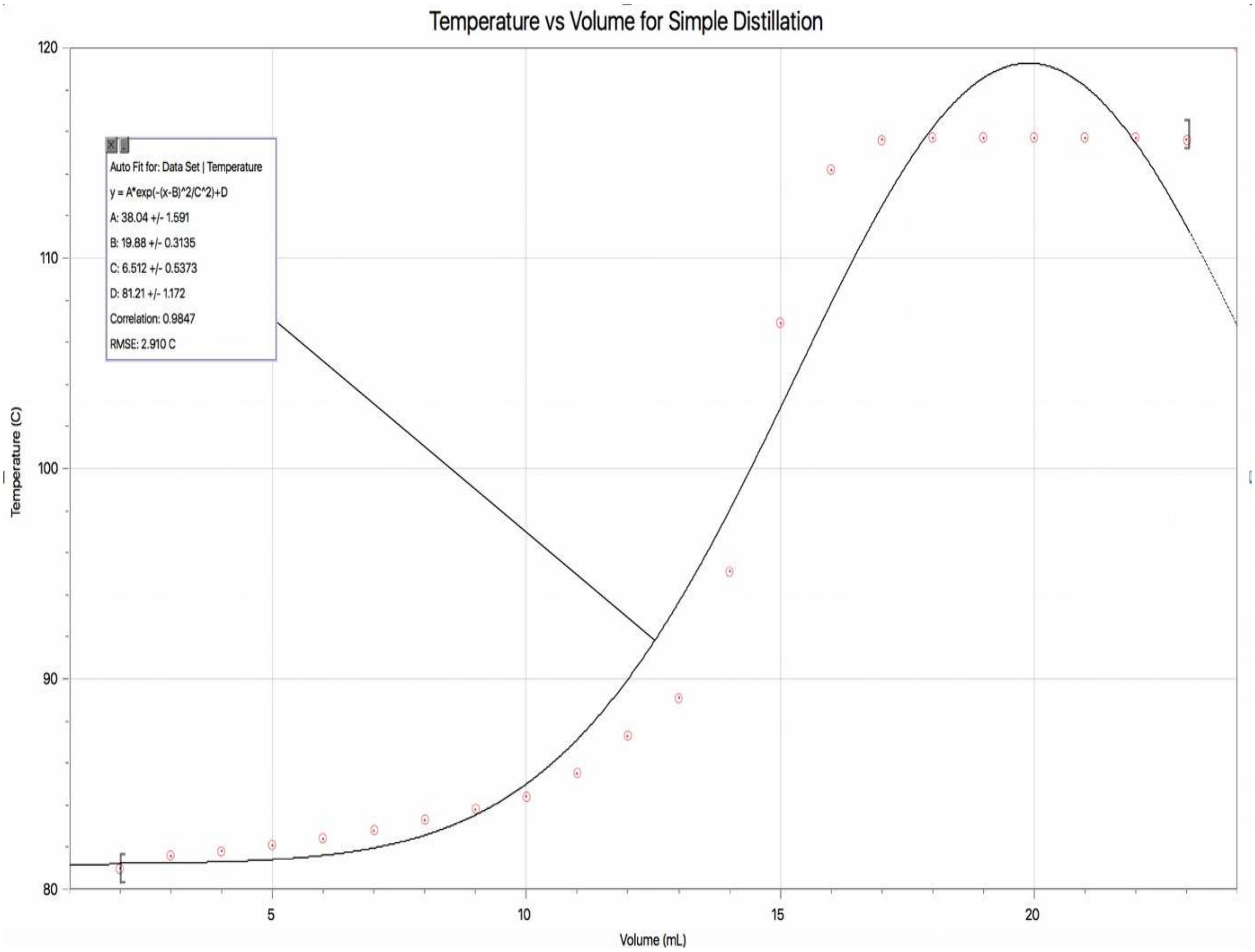


Figure 3 – Temperature (˚C) vs Volume (mL) collected in receiving flask in fractional distillation with Gaussian curve of best fit

***Figure 3 data was borrowed from**

Discussion:

- To begin the discussion, there is obviously significant error in the data collected in the fractional distillation. The quadratic curve in the simple distillation (as seen in figure 1) shows decent results, but figure 2 makes it clear that the fractional distillation did not go as expected.
- Figure 3 is data borrowed by another group in the lab who achieved better results. This will provide a comparison and a Gaussian curve was used as the curve of best fit.
- In the simple distillation, figure 1, there was initially a calibration error with the thermometer when the distillation began that occurred during the 1 mL to 3 mL range. Due to this, temperature values could not be recorded for these volumes,

but would have likely followed the same pattern as seen between 4 to 7 mL just at lower temperatures.

- Even with this, it can be seen throughout the graph that there is generally a constant increase in temperature. Between 4 mL and roughly to 15 mL, the component with the lowest boiling point, 2-propanol, is expected to be dripping into the graduated cylinder. However, it is very likely the two components are not being separated very well, so it can be expected that 1-butanol is also part of this volume the higher the temperature rises.
- At 20 mL, the heat supply was turned off in order to have the apparatus cool down for the following distillation. This should not have been done as it leads to an immediate decrease in temperature. Shortly after there was a very high spike in temperature which was then followed by a plummet in temperature. Although, this does not affect the quadratic curve to a great degree, the curve would have been slightly flatter at the end if this did not happen. This flattening off would of indicate that majority of the 1-butanol has been distilled and the completion of the distillation.
- The simple distillation makes it unclear at what exact point does the 1-butanol actually begin to vaporize as the distillation is nearly linear (maintains the same increasing trend) throughout the entire experiment. This indicates that the simple distillation isn't very effective at separating the two components.

- In the fractional distillation, figure 2, the points were very scattered, not really following the expected trend. They seemed to form two peaks where temperature reaches a maximum with a trough in between where temperature reaches a minimum. It is likely there is error with the heating device as it would be the best explanation for the temperature drop. The heating device must have turned off around the peak temperature then turned back on at the lowest temperature.
- There could have been other factors affecting this as well, such as the possibility of flooding occurring due to over stirring. Although the stirring was kept at no more than 60% of its potential, the column and distilling flask were covered in aluminum, so if flooding did occur, it was not noticed. It is also possible that the apparatus had not cooled down enough, which could have caused the initial spike or there could have also been an opening that allowed heat to escape the distillation apparatus.
- The quadratic curve of best fit makes it look very similar to the simple distillation except it has less of a steep slope. It is clearer where the distillation finishes as the end is flattened out. However, it still looks very similar to figure 1, demonstrating that the collected for figure 2 did not go as expected. However, the difference between figure 2 and figure 3 (the borrowed data) is very evident as figure 3 does not follow a near linear fashion as seen in both figure 1 and 2.
- Analyzing the expected graph for the fractional distillation, it is clear at the beginning that only the 2-propanol is being vaporized at a near constant temperature and falling into the graduated cylinder.
- Following this, there is a very clear spike in temperature that makes it obvious when the 1-butanol has begun to be distilled into the graduated cylinder. The

flattening near the end of the graph indicates that both components have been completely distilled.

- Although the simple and fractional distillation apparatus are very similar, the fractionating column has a very significant impact on the outcome. The fractionating column, due to the addition of its surface area, allows the repeated cycles of vaporizing and condensing of the 1-butanol. This increase surface area between the condenser and distilling flask allows the 1-butanol to fall back down into the distilling flask. The 2-propanol overtime vaporizes all the way up to the condenser due to its lower boiling point and falls into the graduated cylinder.
- This leads to a very effective separation and the distilling flask will only contain 1-butanol (therefore half the volume of the original solution should be left in the graduated cylinder only containing 2-propanol). Once the temperature spikes up and hits a critical temperature (as seen in the centre region of figure 3), the remaining 1-butanol is distilled into the graduated cylinder as well.
- Overall, the fractional distillation apparatus is much more effective at separating the components than the simple distillation apparatus. There was significant error in the collected data, mainly in the fractional distillation of figure 2. However, the data for figure 3 that was borrowed provided data with minimal error in order to analyze and compare in this discussion.
- In future, if time permits, more trials should be taken as the experience from one trial will allow one to perform the following in a better manner. The apparatus should be given sufficient time to cool down after the simple distillation before using it for the fractional distillation. The heating device should be calibrated to ensure that it continually provides heat without interruption and that it provides sufficient heat. The entire apparatus should be inspected very carefully to ensure there is no openings that heat or vapor could be escaping to the environment through.

Questions:

1)

- Liquid must flow back through the fractionating column because after it boils in the flask (turning into vapor), it condenses onto the surface area of the fractionating column due to cooling from travelling up the column.
- The component of the mixture with the higher boiling point will condense onto the column and fall back down into the distilling flask. However, the component with the lower boiling point will keep travelling further up the column into the graduated cylinder.
- The reason this happens is because as the temperature is slowly increased, only the component with the lowest boiling point can actually travel up the surface area of the column to reach the condenser before turning back into liquid. The condenser, due to its cold temperature, turns it into liquid that drips into the graduated cylinder.
- This process repeats itself as cycles of vaporizing and condensing until the substance left in the distilling flask is pretty much only the component with the

higher boiling point. This method of allowing the liquid to drip back into the column provides an effective method in separating the components.

2)

- If a uniform temperature inside the fractionating column is not maintained, it is likely there will be phase changes inside the column that will affect the proper separation of the components.
- The component must evaporate and travel up the column to the condenser to drip into the graduated cylinder. If the temperature is too cold at the top, the vapor of the more volatile component is likely to condense before reaching the condenser and drip back down into the distilling flask. If the temperature is too hot at the top, the vapor of the less volatile component is likely not to drip back down the flask, but rather reach the condenser and mix with the other component in the graduated cylinder.
- Without a uniform temperature gradient in the column, there will not be an effective separation of the components.

3)

- The vapor pressure of benzene at its boiling point is equivalent to that of atmospheric pressure. Therefore, it is equal to 1.01325 bar or 760 mmHg.

4)

- The boiling point of a liquid will increase as the atmospheric pressure is increased. This is due to the liquid only being able to become vapor if its vapor pressure overcomes atmospheric pressure.

5)

- By having water enter through the bottom of the condenser, the entire condenser would be filled with water before exiting through the exit near the top. This allows proper cooling of the vapour by the entire condenser.
- If the water were to enter from the top, would let less water be present in the condenser due to gravity pushing it faster down the condenser to the exit (which would probably be at the bottom). This would make only part of the condenser effective as not all the condenser would be filled.
- There is also the risk of the glass cracking if the water was filled from the top end of the condenser. The coldest point is at the bottom of the condenser would filled by the bottom, so this prevents the glass from possibly breaking due to the heat of the vapor in contact with the low temperature of the condenser.

6)

Mole fraction A = $\frac{3}{3+1}$ = 0.75

Mole fraction B = $\frac{1}{3+1}$ = 0.25

P_{Total} = (Mole fraction A)*(Vapor pressure A) + (Mole fraction B)*(Vapor pressure B)

= (0.75)*(350 mmHg) + (0.25)*(150 mmHg)
= 300 mmHg

- Therefore, the vapor pressure of the mixture at 95˚C is 300 mmHg.

Acknowledgements:

- Figure 3 data was borrowed by
- Figure 1 and 2 were made using Logger pro application.