2. Purifying Chemicals by Distillation

Name
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CHM1311

Demonstrator
Partner

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Department of Chemistry
University of Ottawa

Introduction
In this experiment we will be using two different methods of distillation in order to purify compounds. Distillation is a process of purification where a mixture of substances are boiled. The two substances will evaporate at their respective boiling points and then get cooled and condensed back into liquid form in a separate flask, now in purified form.

Most pure substances have their respective boiling points that span a few degrees - this is a way of identifying substances. The more polar the substance, the more hydrogen bonding and therefore a higher boiling point. Substances low in polarity/non-polar will have weaker bonds between atoms and will therefore boil at lower temperatures. As a general rule, the larger the molecule, the higher the boiling point.

The first distillation that will be performed will be a simple distillation. In a simple distillation, the substance to be distilled is put into a flask where it is constantly stirred and heated slowly. The vapour then travels up past the thermometer before being redirected through the condenser, where the cooled glass will allow the vapour to change back into liquid form. As the vapour condenses, the liquid will slowly drip out of the condenser and into the receiving flask. When purifying a mixture, the receiving flask can be changed out throughout the experiment to collect different portions of the distillate. For example, if a mixture of two substances is being distilled, changing the flask in between the two boiling points of the substances will result in the first flask being mostly the substance with the lower boiling point, and the second flask will be mostly composed of the substance with the higher boiling point. Since only mixtures will be purified in this experiment, we will see a constant increase in temperature as the distillation proceeds. Simple distillation is best used to purify a substance which is already mostly composed of only one substance, or when purifying a mixture which has a large difference in boiling points.

Raoult's law is an equation which shows that when boiling a mixture, there will be more of the lower boiling point substance's vapour than there will be of its liquid, meaning that the substance will the lower boiling point will get vaporized first, leaving the rest of the liquid mostly composed on the liquid from the substance with the higher boiling point. This equation is:

$$Total\ pressure = (PA)(NA) + (PB)(NB)$$

Where PA/PB is the partial pressure of substance A or B, and NA/NB is the mole fraction of substance A or B in the mixture. As the distillation proceeds, the substance with the lower boiling point will get removed by vapourizing, and the liquid remaining in the distilling flask will be composed mostly of the substance with the higher boiling point.

Fractional distillation is the second type of distillation used in this experiment. This type of distillation is very similar to the simple one with the exception that fractional distillation has a fractionating column. Fractionating columns are ones which have been packed with a material to create a large surface area. The purpose of this column is so that the vapour can condense and vapourize many times. This separates out the distillate even more than the simple distillation since the vapour is able to condense and drip back down into the distilling flask if it contains more of the substance with the higher boiling point, or it can be vapourized again if it contains mostly the substance with the lower boiling point. This will be done many times in the fractionating column to better separate out the components.

In this experiment, a 1:1 mixture of 1-butanol and 2-propanol will be distilled. 2-propanol has a boiling point of 83C, making it more volatile, while the 1-butanol has a boiling point of 118C.

Procedure

As described in the CHM1321 Organic Chemistry lab manual.

1. Glassware was set-up and approved before each distillation.
2. During the fractional distillation, the temperature reached 117C and stayed there. Since the distillate was not vapourizing, the temperature was increased slightly and tin foil was added to insulate the distilling flask.
3. After experiment was complete, mixture was disposed of into organic waste, glassware was cleaned and put away.

Observations

- About halfway through each distillation, the liquid in the receiving flask began to look ‘squiggly’ as liquid dripped in. This could be due to the different purified substances in the mixture mixing together slowly.
- The 1:1 mixture 1-butanol:2-propanol was clear in colour.
- The simple distillation was very quick, with a mostly linear steady increase in temperature throughout.
- The fractional distillation took some time to complete, about halfway through the distillation, the temperature shot up very quickly.
- The temperature probe would collect a drop of mixture on the tip as the mixture vapourized. When the drop would fall off, there was a slight decrease in temperature for a second or two, before stabilizing.

Data Tables/Graphs

Table 1: Simple distillation. Temperature was noted after each mL dripped out into receiving flask.

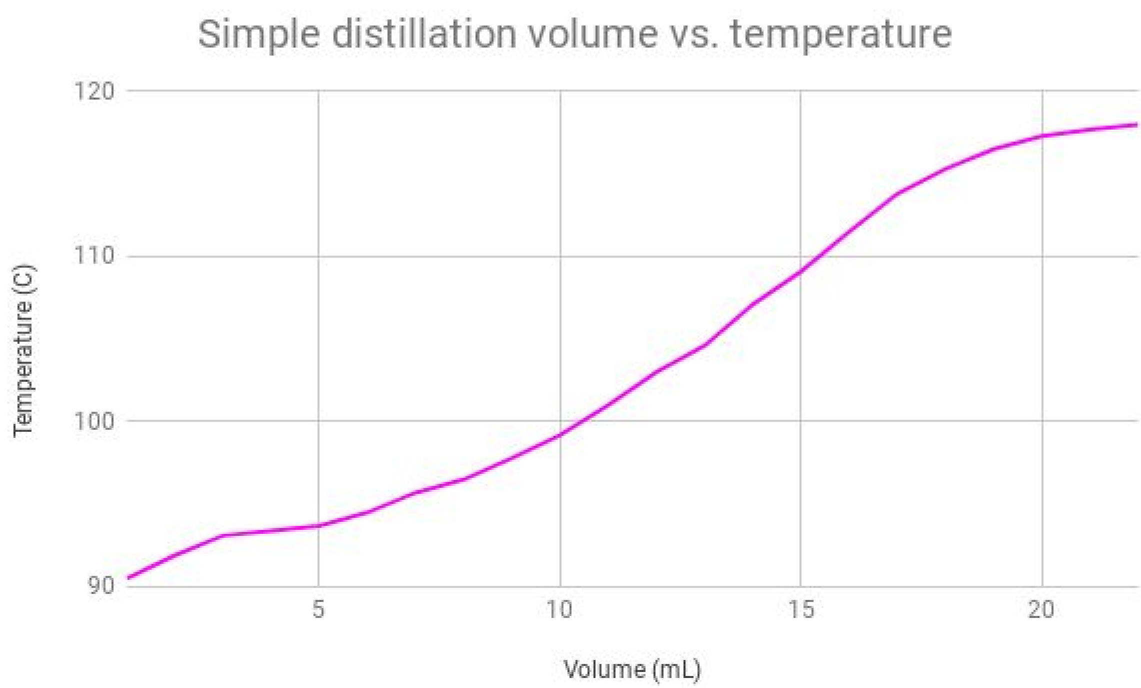
Volume (mL)	Temperature (°C)
1	90.5
2	91.9
3	93.1
4	93.4
5	93.7
6	94.5
7	95.7
8	96.5
9	97.8
10	99.2
11	101.0
12	103.0
13	104.6
14	107.1
15	109.1
16	111.5
17	113.8
18	115.3
19	116.5
20	117.3
21	117.7

22	118.0

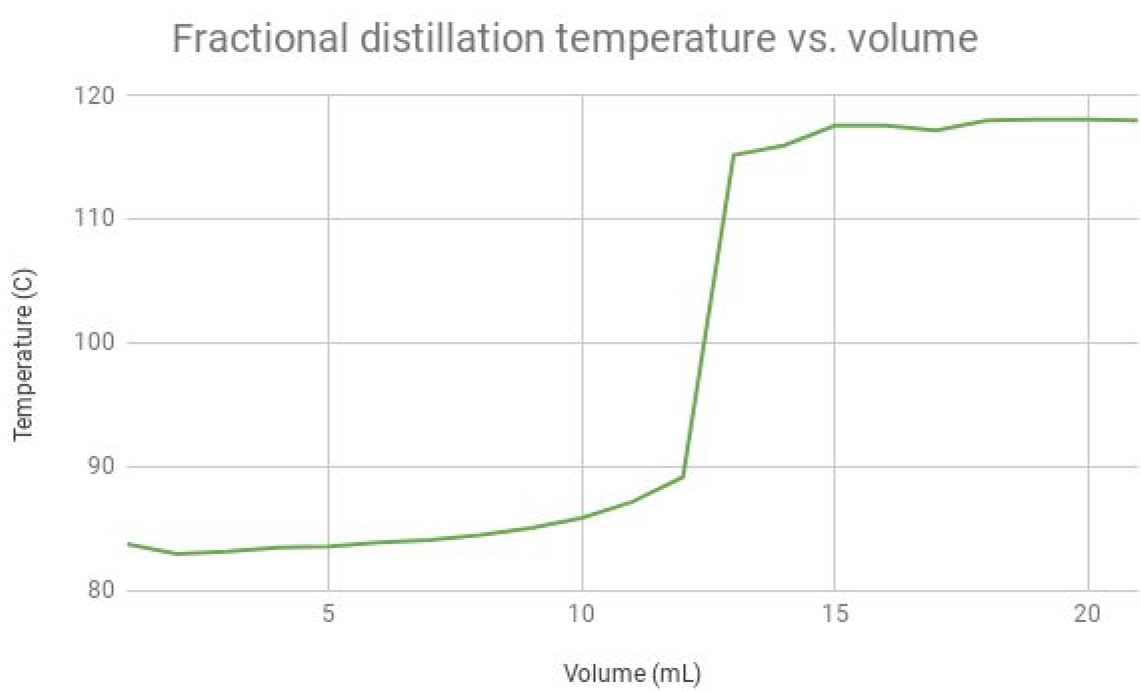
Table 2: fractional distillation. Temperature was recorded after each mL dripped out into receiving flask.

Volume (mL)	Temperature (°C)
1	83.8
2	83.0
3	83.2
4	83.5
5	83.6
6	83.9
7	84.1
8	84.5
9	85.1
10	85.9
11	87.2
12	89.2
13	115.2
14	116.0
15	117.6
16	117.6
17	117.2
18	118.0
19	118.1
20	118.1
21	118.0

Graph 1: simple distillation graph plotting temperature vs. volume.



Graph 2: fractional distillation graph plotting temperature vs. volume.



Questions

1. Explain why you must have liquid flowing back through the fractionating column in order to get separation of the components during a fractional distillation.
    a. Fractional distillation works by having the distillate get vapourized and condensed many times on the fractionating column so that a better separation occurs. When some of the mixture gets vapourized, it contains both of the substances within the mixture, not just the lower boiling point substance. When the mixture then condenses on the packing, the 1-butanol will drip back down into the distilling flask because it has a boiling point of 118C, while the 2-propanol will get vapourized again, and will make its way through the condenser where it will get re-condensed and drip into the receiving flask. This occurs because 2-propanol has a lower boiling point and will therefore get purified before 1-butanol. Fractional distillation is efficient because it technically does many distillations in the fractionating column to better separate out the mixture.
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?
    a. It is important to maintain a smooth temperature gradient in a fractionating column because as the mixture vapourizes, it will move into the condenser to get collected. If the temperature is fluctuating it will be harder to get a good separation, distillate will vapourize at one temperature and condense if it lowers, making the distillation inefficient.
3. The boiling point of benzene is 81 °C. What is the vapour pressure of benzene at this temperature?
    a. As stated in the manual, the vapour pressure of benzene at this temperature would be equal to atmospheric pressure since benzene would be a pure liquid.
4. What effect does an increase in atmospheric pressure have on the boiling point of a liquid?
    a. Increasing the atmospheric pressure would also increase the boiling point, lowering pressure would lower the boiling point. This is because the vapour pressure is equal to the force applied.
5. Why is it important to have cooling water enter the bottom of the condenser and not the top?
    a. If the water enters from the bottom of the condenser, the jacket will fill up with water (as seen in lab), if the water enters from the top, it tends to flow

down one side only. For the distillate to condense, it must be exposed to cool glass. Condensing will be less efficient if only half of the condenser is cooled.

6. Compound A has a vapour pressure of 350 mm Hg at 95 °C whereas compound B has a vapour pressure of 150 mm Hg at the same temperature. If A and B are miscible, what is the vapour pressure of a 3:1 mixture of A and B at 95 °C?
    a. $Total\ pressure\ =\ (PA)(NA)\ +\ (PB)(NB)$
       P = (350mmHg)(0.75)+(150mmHg)(0.25)
       P = 262.5 + 37.5
       P = 300mmHg
       Therefore the vapour pressure will be 300mmHg.

Discussion

As shown in the lab manual, the temperature curve for a simple distillation was a gradual increase. Graph 1 shows that our simple distillation followed this pattern. This is how we expected the graph to look. Since we were distilling a mixture of substances, the temperature gradually increased as it purified both compounds. Near the beginning, mostly 2-propanol was being vapourized, then the temperature increased steadily until it levelled out slightly as the boiling point for 1-butanol was reached, where mostly 1-butanol was being purified.

The curve for the fractional distillation was also similar to the one shown in the lab manual. This curve (graph 2) was mostly constant as the boiling point for 2-propanol was reached. At this point, 2-propanol was being purified and condensed. After most/all of the 2-propanol was collected, the temperature quickly increased to the boiling point for 1-butanol, where it levelled out again as the 1-butanol was being collected. This is how we expected the curves to look since fractional distillation is more efficient/better at separation than simple distillation.

The fractional distillation was the better choice for purification for this mixture. As seen in the graphs, it is obvious in graph 2 when 2-propanol and when 1-butanol were being collected based on the temperature. Since there was also only a 35C difference in melting points, this method was more useful. Had the differences been over 100C different, the simple distillation would have probably been a good choice - it is easier to set up, less glassware is required, and less objects to clean etc.

A possible source of error in this experiment could have been that in the fractional distillation, the temperature probe would show that the temperature changed slightly as the drop of mixture fell off the tip (approximately 0.5-1C difference). Care was made to record the temperature after it stabilized but this could have affected the accurate reading of the thermometer.

Conclusion

In conclusion, the fractional distillation was a better method of purification for this mixture.

Bibliography

1. CHM1321 organic chemistry lab manual
2. http://www.pitt.edu/~ceder/lab7/fractionaldistillation.html

Raw Data