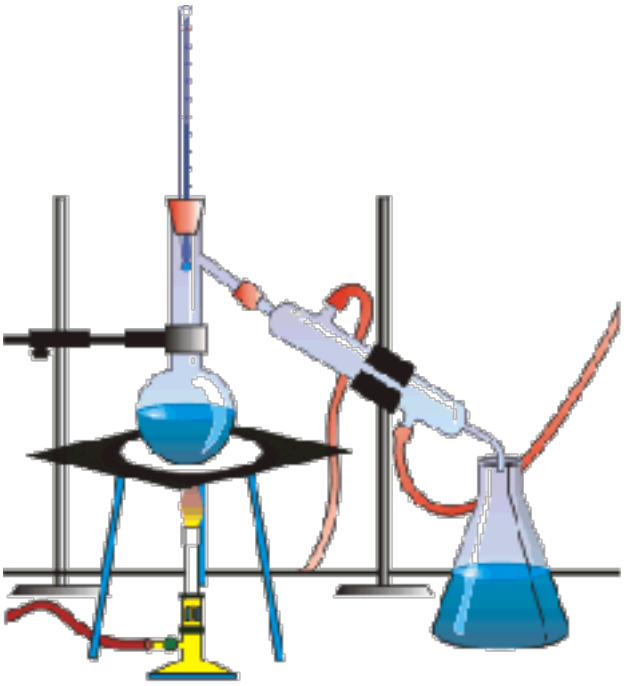
Simple and Fractional Distillation

Purifying Chemicals



Procedure:

As described in the Organic Chemistry Laboratory Manual on pages 25 to 27. However, one modification was made; instead of using the 100mL distillation flask, the 50mL distillation flask was used.

Observations:

- 50:50 2-propanol and 1-butanol: This was a colourless, transparent liquid with a strong odour that resembled nail polish remover
- The distillate had the same appearance/smell as the original solution, after each distillation
- The temperature was increasing at a steady rate during the simple distillation
- The temperature of the second distillation was increasing very slowly, jumped rapidly by approximately 20℃ after 11mL of distillate, then continued to level out

Tables:

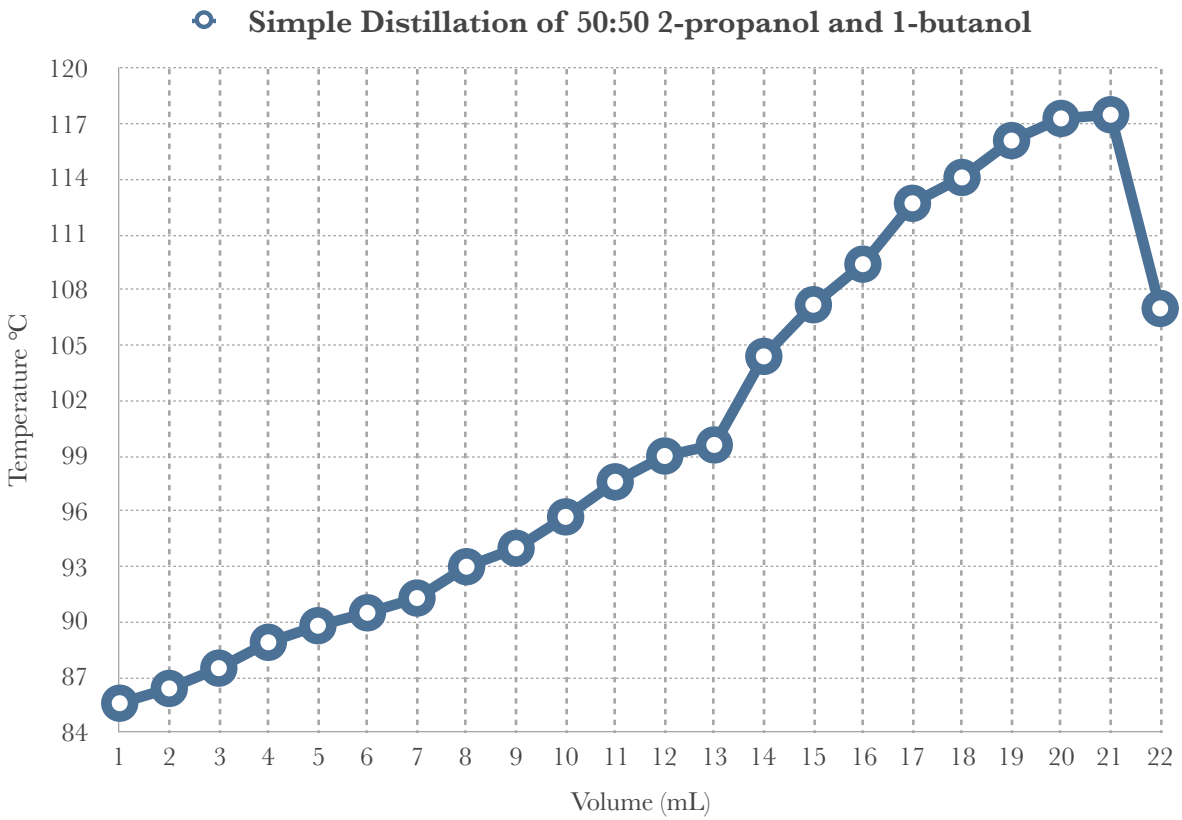
Table 1: **Simple Distillation of 50:50 2-propanol and 1-butanol**

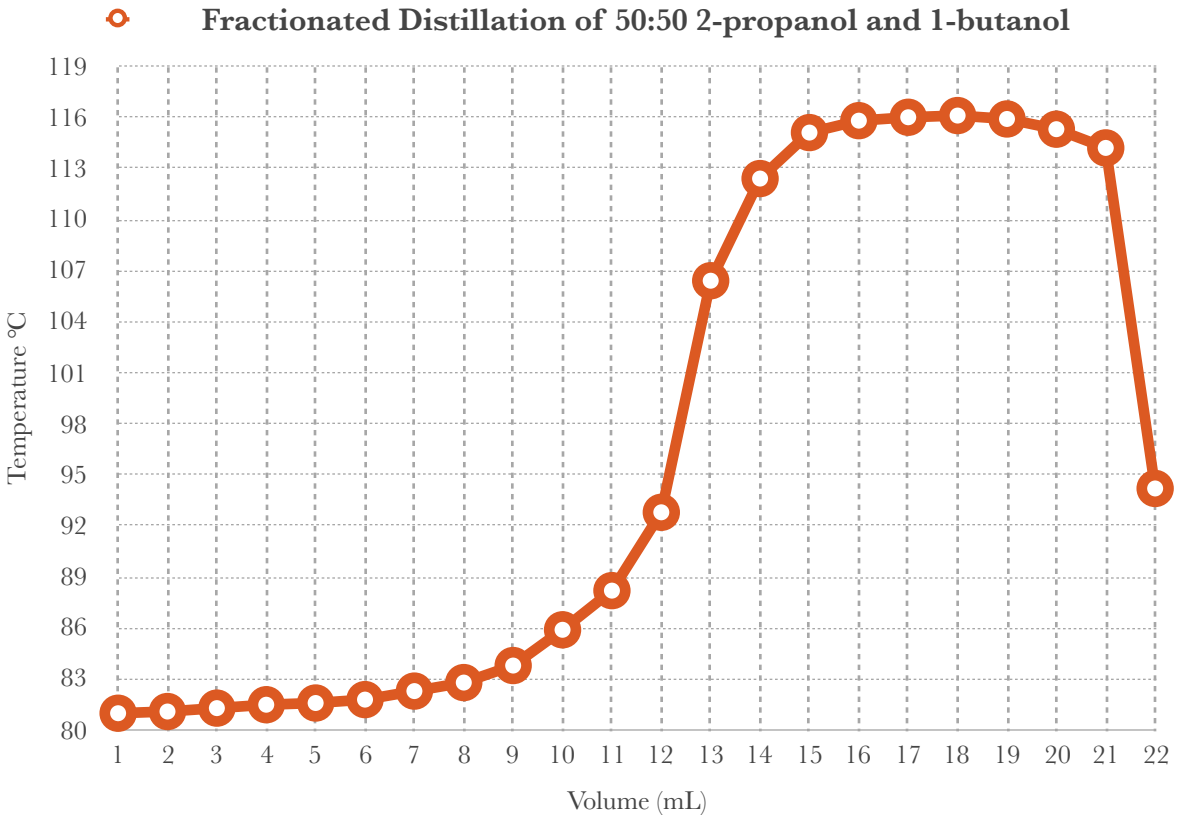
Volume of 50:50 2-propanol and 1-butanol distillate (mL)	1	2	3	4	5	6	7	8	9	10	11
Temperature (℃)	85.6	86.4	87.5	88.9	89.8	90.5	91.3	93.0	94.0	95.7	97.6
Volume of 50:50 2-propanol and 1-butanol distillate (mL)	12	13	14	15	16	17	18	19	20	21	22
Temperature (℃)	99.0	99.6	104.4	107.2	109.4	112.7	114.1	116.1	117.3	117.5	107.0

Table 2: **Fractioning Distillation of 50:50 2-propanol and 1-butanol**

Volume of 50:50 2-propanol and 1-butanol distillate (mL)	1	2	3	4	5	6	7	8	9	10	11
Temperature (℃)	81.0	81.1	81.3	81.5	81.6	81.8	82.3	82.8	83.8	85.9	88.2
Volume of 50:50 2-propanol and 1-butanol distillate (mL)	12	13	14	15	16	17	18	19	20	21	22
Temperature (℃)	92.8	106.4	112.4	115.1	115.8	116.0	116.1	115.9	115.3	114.2	94.2

Graphs:


Simple Distillation of 50:50 2-propanol and 1-butanol
Temperature °C
120
117
114
111
108
105
102
99
96
93
90
87
84
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22
Volume (mL)


Fractionated Distillation of 50:50 2-propanol and 1-butanol
Temperature °C
119
116
113
110
107
104
101
98
95
92
89
86
83
80
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22
Volume (mL)

Discussion:

For the experiments, the temperature of the heating mantle was originally set to 60℃ then increased to 90℃ to speed up the distillations. During the simple distillation, the temperature increased at a slow and steady rate; this was due to the fact that there was not a large surface area for the separation of 2-propanol and 1-butanol to occur. When the fractionating column was added between the distillation head and the distillation flask, a large temperature change was noted, which indicates a composition change. This means that overall the fractional distillation was more effective than the simple distillation at separating the mixture. The fractionated column contained packing (metal sponge or glass wool) which increased the surface area and gave the vapour somewhere to condense on while the distillation progressed. The fractionated column also had the ability to re-vaporize some of the condensate.

2-propanol has a lower boiling point than 1-butanol, thus making it more volatile and less stable. During the distillation 2-propanol would have condensed first and ended up in the receiving flask first. 1-butanol stayed in the distilling flask as a liquid for a longer period of time. Once all of the 2-propanol had been separated, the 1-butanol could vaporize, travel up the fractionating column, travel through the condenser where it cools and returns to it's liquid form, then drip into the receiving flask.

According to Raoult's law, the vapour will contain more of the liquid with a high vapour pressure (liquid with a lower boiling point) than is present in the liquid. In other words, the liquid solution is composed of 50% 2-propanol and 50% 1-butanol, but the vapour will have a higher percentage of 2-propanol. The differences in boiling points is what causes the two solutions to distillate at different rates. As the concentration of 1-butanol increases in the mixture, the temperature needed to create vapour must increase as well.

Using the magnetic stirrer and stir plate was important, especially during the second distillation. Since some separation would have occurred, and the solution needed to be topped up, the composition of 50:50 2-propanol and 1-butanol would not have mixed otherwise.

Due to the fact that simple distillation is not as effective as fractioning distillation, it was expected that the graph would be linear and have a positive slope. The steady rate of change at the beginning of the graph represents the vaporization of 2-propanol. At about 14mL of distillate the temperate rose, which means that the 1-butanol was also beginning to vaporize. When looking at the second graph, there is a S shaped curve rather than a straight line. From 1mL to 8mL of distillate, the 2-propanol was vaporizing because it has a lower boiling point. The steep slope from 9mL to 14mL is when most of the 2-propanol had separated from the mixture and the 1-butanol could begin to vaporize. From 15mL to 21mL of distillate 1-butanol, the solution with a higher boiling point, was vaporizing. The reason that the rapid increase in temperature is more

noticeable in graph 2 was because of the fractionating column, which allowed for better separation of the mixture. 2-propanol has a boiling point of 86.2℃ which matches the temperature of the first section of the curve. Then the line makes a sharp incline to reach the boiling point of 1-butanol, which is 117.7℃. These numbers are very similar to those recorded during the experiment which indicated that separation and distillation was in fact occurring.

At the end of the graphs, 22mL of the distillate, the temperature significantly dropped each time. This occurred because there was no more solution left in the distilling flask, so we knew the distillation had to be over. Technically, these values are not important and do not need to be included in the analysis of the data.

Sources of Error

In this experiment, the setup of the equipment was critical. If any part of the apparatus was placed incorrectly, the distillation would not work or the data would be skewed. Especially in the case of the water in/out tubes, if they were not on tight enough, one could pop off and water would spray everywhere. Similarly, vapour could escape at the junctions, so they had to be sealed tightly. Flooding was also something that had to be avoided. Human error could be a contributing factor in this experiment. It was possible that the temperature was recorded slightly before/after every 1mL of distillate, which would slightly change the shape of the graphs. A piece of equipment that could tell you the temperature after exactly every 1mL would be very useful and aid with precision.

Questions:

1. Liquid flowing back through the fractionating column means that the boiling point of only one component has been reached, which implies there is an effective separation of the two components. The compound with the higher boiling point, 1-butanol, will drip back down the fractionating column because it does not have enough energy to stay in the vaporized state. Consequently, it does not reach the condenser, and will turn back into liquid on the metal sponge or on the column, then drip back into the distilling flask. No liquid flowing back through the fractioning column would indicate that the temperature was high enough for both compounds to vaporize and reach the condenser, meaning no separation has come about.
2. Maintaining a uniform temperature gradient in a fractioning column is critical for ensuring the the vapour makes it to the receiving flask. If it temperature in the column decreases before the vapour reaches the condenser, all the liquid would remain in the distilling flask and no separation would occur. If the temperature was fluctuating between the initial temperature and the temperature in which the less volatile solution boils, then the components would not correctly separate because some of the 1-butanol would join the vapour from the 2-propanol. Therefore, the temperature gradient is the key to a successful distillation.
3. The boiling point of a liquid is reached when the vapour pressure is equal to the atmospheric pressure. Therefore, when benzene reaches a temperature of 81℃, the vapour pressure will be 1.0atm or 760mmHg.

4. Increasing the atmospheric pressure would cause the boiling point of a liquid to increase as well. When the surrounding pressure is increased, the vapour pressure increases concurrently which means that a greater amount of energy would be required to boil the liquid.
5. It is important to have the cooling water enter at the bottom of the condenser and not the top because we want the vapour to go through the condenser and turn to liquid just prior to entering the receiving flask. If the cooling water entered at the top then the cold temperature could cause the vapour to condense sooner and drip back into the distilling flask, which would inhibit the separation of the compounds. Also, putting the cooling water at the bottom ensures that the area around the condenser allowed for water is completely full and evenly distributed.
6. According to Raoult's law, the vapour pressure of a 3:1 mixture of compounds A and B is 300 mmHg. [(0.75 x 350) + (0.25 x 150)]

Raw Data: