Experiment 2: Purifying Chemicals by Distillation

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Procedure: Refer to *Introductory Organic Chemistry: Laboratory Manual*, pages 20-27.
Observations and results:

- 50:50 mixture of 2-propanol and 1-butanol: clear, colourless, aqueous solution; no discernible odour
- Magnetic plate set to 7
- Voltage on heating plate set to 70

Table 1 – Data collected from simple distillation

Temperature (°C)	Volume (mL)	Temperature (°C)	Volume (mL)
83.7	1	96.7	11
86.1	2	99.5	12
86.8	3	102.8	13
87.7	4	106	14
88.4	5	109.4	15
89.5	6	112.5	16
90.6	7	115.4	17
91.8	8	116.4	18
93.5	9	116.9	19
95	10	117.2	20

Graph 1 – Volume of distillate (mL) collected from simple distillation as a function of temperature (℃)

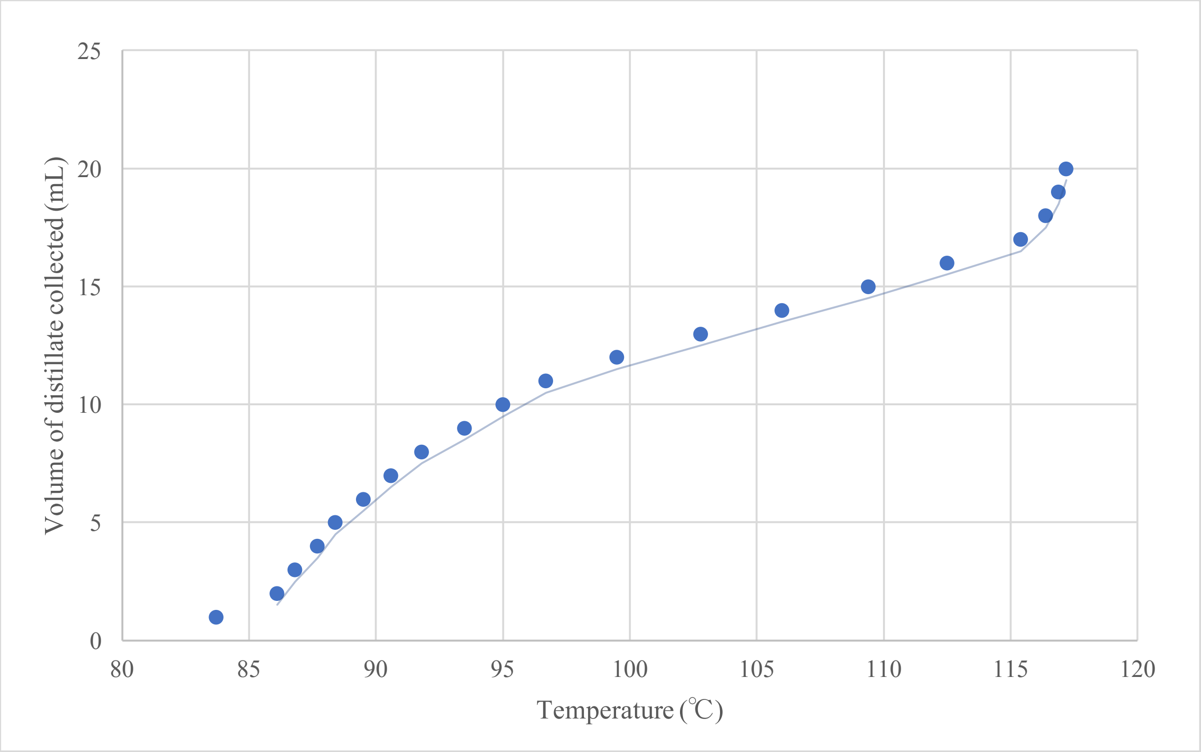
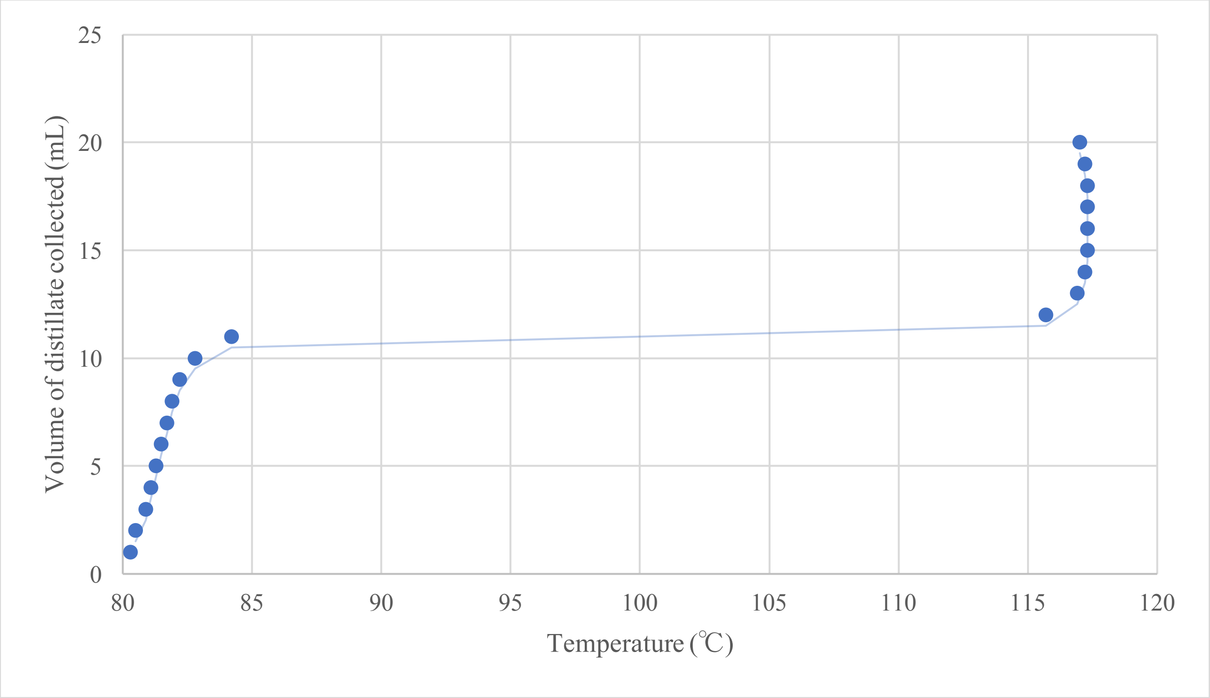


Table 2 – Data collected from fractional distillation

Temperature (℃)	**Volume (mL)**	**Temperature (℃)**	**Volume (mL)**
80.3	1	84.2	11
80.5	2	115.7	12
80.9	3	116.9	13
81.1	4	117.2	14
81.3	5	117.3	15
81.5	6	117.3	16
81.7	7	117.3	17
81.9	8	117.3	18
82.2	9	117.2	19
82.8	10	117	20

Graph 2 – Volume of distillate(mL) collected from fractional distillation as a function of temperature (℃)



Discussion:

- Both simple and fractional distillations were conducted for the same mixture in order to compare their relative efficiencies at separating the distillate

- The distillate was a 50:50 mixture of 2-propanol and 1-butanol.
    - The boiling point of 2-propanol is 82.6°C, the boiling point of 1-butanol is 117.7°C.
    - The difference in the boiling points of the two components is not very significant! (35.1°C)
    - 1-butanol is less volatile due to its larger size, exhibiting stronger Van der Waal's forces. 1-butanol is also a primary alcohol, meaning that the hydroxyl group is more "available" to engage in hydrogen bonding; this also accounts for its higher BP.
- The purpose of both processes is to separate the mixture on the basis of the boiling points of its components. Both simple and fractional distillations are often used in industrial settings to purify compounds.
- Both distillations required a similar apparatus, with the addition of a fractionating column for the fractional distillation.
- From the relative steeper slope of graph 1, it is evident that simple distillation occurred more quickly than the fractional distillation, whose slope (shown in graph 2) is more gradual.
- Graph 1 cannot be divided into two distinct regions (as the graph 2 is) indicating a less effective separation of the mixture.
    - In the simple distillation, the relation between volume of distillate collected and time is very gradual.
- The long horizontal portion of the graph 2 indicates a slower but more effective separation of the mixture for simple distillation.
    - This portion of the graph represents a period of time where temperature is increasing steadily, but the volume of distillate collected remains constant.
    - The horizontal portion of the graph began at 84.2 °C and ended at 115.7 °C; these values correspond to the temperature at which all the 2-propanol had been removed, and when the 1-butanol began to vapourize.
    - 2-propanol began vapourizing at 80.3°C (2.3 °C below its BP) and 1-butanol vapourized at 2 °C lower than its BP. This could be due to the formation of an azeotrope. Azeotropes form when two liquids exhibit a positive deviation, causing lower boiling points than anticipated. The differing BP's may also be attributed to impurity of the liquids.
    - As the sample repeatedly vapourizes and condenses, more of the more volatile component (2-propanol) is removed from the first flask and condensed into the second.
    - At 84.2°C, all of the 2-propanol had been removed from the first flask; vapourized and condensed into the second!
    - At 115.7 °C, the 1-butanol began to vapourize.
- From the results obtained, it can be confirmed that fractional distillation is more effective when separating a mixture of two components with similar boiling points
    - This is because simple distillation vapourizes and condenses the distillate only once, whilst fractional distillation functions by vapourizing and condensing the

distillate multiple times.

Sources of error, strengths, and potential expansions

- At the end of the fractional distillation, temperature was held constant (at 117.3 ^{o}C) for a certain period of time, and then fell slightly. This is important to note, as during the entire course of the distillation, temperature should have risen uniformly. However, at this point, there was almost no distillate remaining in the first flask, so this likely had no significant impact on the results.
- The techniques employed were not particularly difficult, and if the procedure was followed closely, the experiment was prone to few sources of error.
- Leaving the connector tube open during both the simple and the fractional distillations ensured that the pressure of the system was equal to atmospheric pressure. This prevented fracturing of the apparatus.
- Success of the experiment depended largely on a sufficient amount of time (and time was a constraint due to malfunctioning thermometers/equipment). Detailed below are some ways to maximize the amount of time given:
- The fractional tube used in the second distillation was insulated with a packing. This ensured a more efficient distillation by minimizing the amount of heat and time required to complete the distillation.
- In future distillations, it might be more efficient to lower the pressure of the system by use of a vacuum. This means that less energy would be needed to reach the boiling point of the liquid, and the experiment could be performed more quickly.

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.

   Fractional distillation functions by repeatedly vapourizing and condensing a sample. Liquid must flow back through the fractionating column in order to obtain a more enriched sample. Without the flow of liquid, the less volatile compound will not condense and return to the first flask; rather, both components will remain vapourized and the separation of the components will be less effective.

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?

   The effective separation of the mixture requires a uniform temperature gradient, and this is best achieved by some form of insulation or packing. The fractionating column must be warmer at the bottom, and cooler at the top. If this gradient is not maintained smooth, then the less volatile component will not condense as needed, and the sample will not be properly enriched. The result of the separation would ergo not yield a distinct separation of the two components of the mixture.

3. The boiling point of benzene is 81 °C. What is the vapour pressure of benzene at this temperature?

   Vapour pressure = atmospheric pressue
   = 101.325 kPa

4. What effect does an increase in atmospheric pressure have on the boiling point of a liquid?

   The boiling point of a liquid designates the temperature at which the pressure applied to the system is equal to the vapour pressure of the solution. As the pressure applied to the system increases, more energy is needed for the liquid to reach its vapourizing pressure. Therefore, as atmospheric pressure increases, boiling point increases.

5. Why is it important to have cooling water enter the bottom of the condenser and not the top?

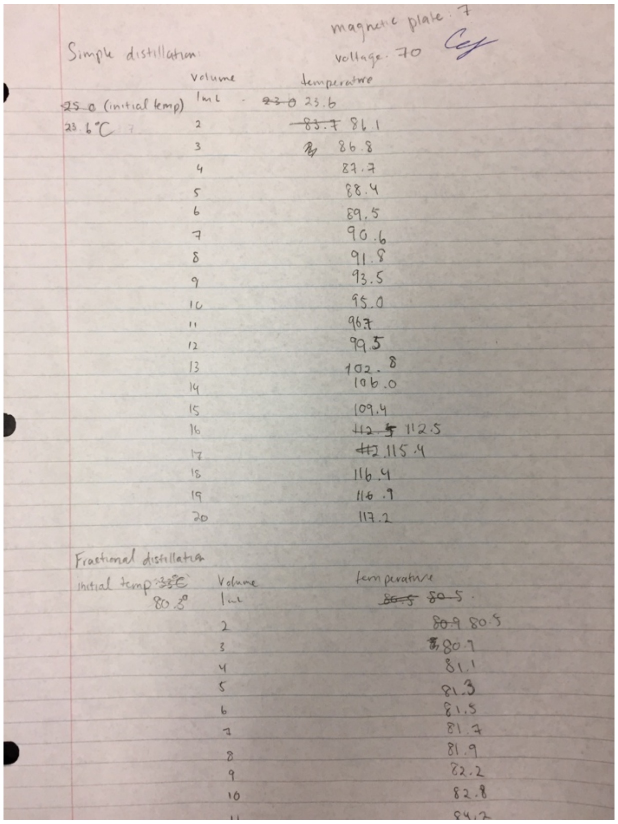
   The condenser is more full when cooling water enters from the bottom, not the top. This is critical since the function of the condenser requires a sufficient amount of water to cool the vapour!

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?

   Using Raoult's Law:

   $$\begin{aligned} P_{\text{Total}} &= (P_{A^\circ})\cdot(N_A) + (P_{B^\circ})\cdot(N_B) \\ &= (350)\cdot(\tfrac{3}{4}) + (150)\cdot(\tfrac{1}{4}) \\ &= 300 \end{aligned}$$

Raw data:



magnetic plate: 7
voltage: 70

Simple distillation:

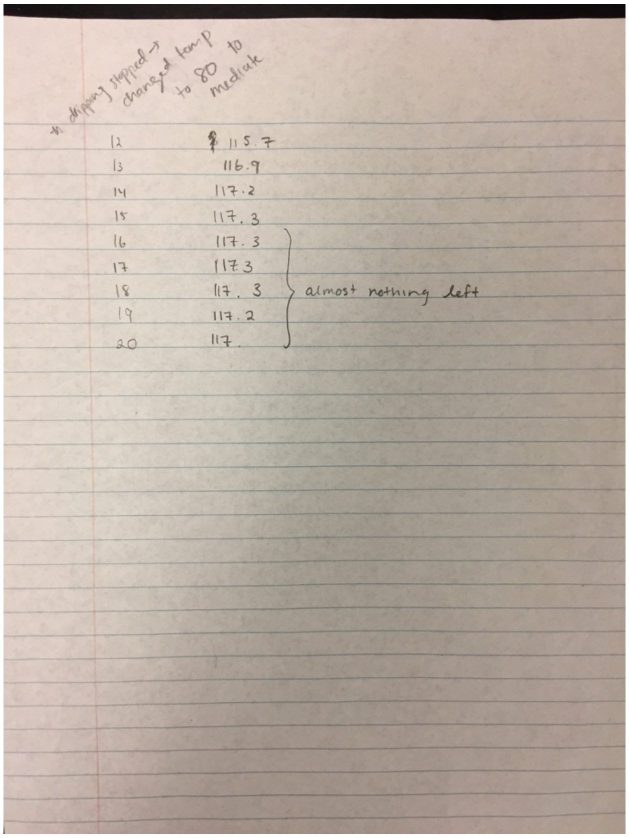
~~25.0~~ (initial temp)
23.6°C ?

Volume	temperature
1mL	~~23.0~~ 23.6
2	~~83.7~~ 86.1
3	86.8
4	87.7
5	88.4
6	89.5
7	90.6
8	91.8
9	93.5
10	95.0
11	96.7
12	99.5
13	102.8
14	106.0
15	109.4
16	~~112.5~~ 112.5
17	~~112~~ 115.4
18	116.4
19	116.9
20	117.2

Fractional distillation

initial temp: ~~33°C~~
80.3°

Volume	temperature
1mL	~~80.5~~ 80.5
2	~~80.9~~ 80.5
3	80.7
4	81.1
5	81.3
6	81.5
7	81.7
8	81.9
9	82.2
10	82.8
11	84.2



* dripping stopped → changed temp to 80 to mediate

12	115.7
13	116.9
14	117.2
15	117.3
16	117.3
17	117.3
18	117.3
19	117.2
20	117.

} almost nothing left