Experiment 2

Purifying Chemicals by Distillation

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Procedure:

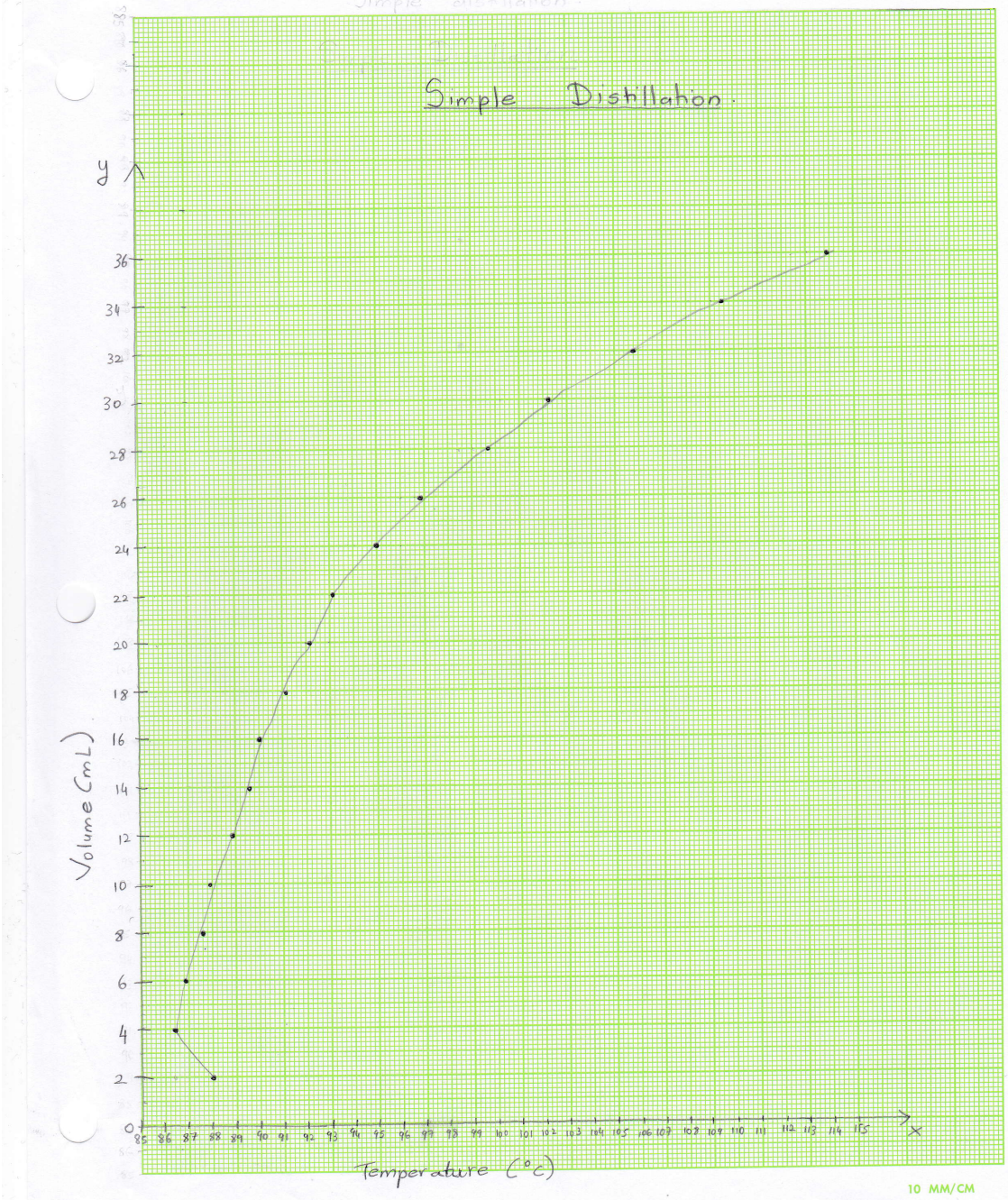
Procedure was followed as mentioned in the lab manual. The graduated cylinder used during simple and fractional distillation was 50mL- not 100mL. During fractional distillation the 100mL distilling flask and the fractionating column were covered with foil for insulation. During both procedures a measuring cylinder was used as the receiving flask.

Observations:

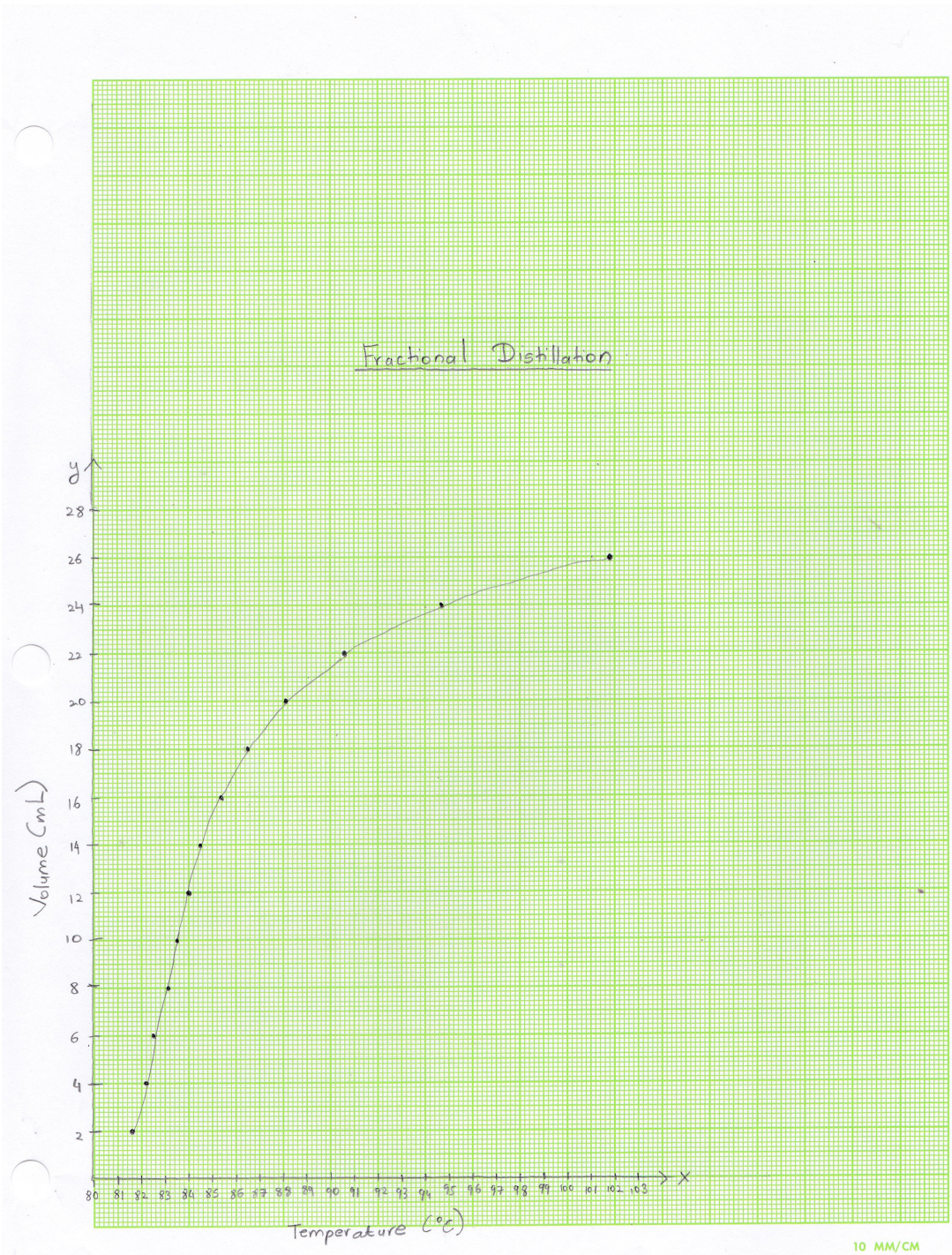
Simple Distillation	**Fractional Distillation**
The 50mL of a 50:50 mixture of 2-propanol and 1-butanol used was clear, transparent, odourless solution. During the process of distillation no bubbles were seen inside the condenser. Bubbles were seen inside the distilling flask as it was heating. The condenser was covered with steam during the procedure. The pattern in which the drops fell to the measuring cylinder was "2 drops and pause, 2 drops and pause…" Initial temperature of the transformer was 80^{o} Celsius and it was increased to a bit over 100^{o}. The very first temperature recorded at 2mL was rounded to digits by the machine. A greasy substance (immiscible) started to drop at 113.9^{o} Celsius after 36mL. The final temperature measured was at 36mL.The drops continued to fall.	Same mixture of 2-propanol and 1-butanol was recycled from simple distillation to fractional distillation. No bubbles seen in the condenser. Steam formed inside the condenser. Bubbles were seen in the distilling flask. The temperature increased slowly initially and then, after about 16mL the temperature increased more rapidly. After about 26mL the greasiness started to appear. The procedure was stopped at this volume. The initial temperature of the transformer was 80^{o} Celsius and was increased to about 100^{o} Celsius (less than the temperature set during simple distillation.

Graphs:

Simple Distillation	
Volume (mL)	**Temperature (℃)**
2.0	88.0
4.0	86.4
6.0	86.9
8.0	87.6
10	87.9
12	88.9
14	89.6
16	90.0
18	91.1
20	92.1
22	93.1
24	95.0
26	96.8
28	99.7
30	102.2
32	105.8
34	109.5
36	113.9


Simple Distillation.
y
Volume (mL)
0
2
4
6
8
10
12
14
16
18
20
22
24
26
28
30
32
34
36
85 86 87 88 89 90 91 92 93 94 95 96 97 98 99 100 101 102 103 104 105 106 107 108 109 110 111 112 113 114 115
x
Temperature (°C)
10 MM/CM

Fractional Distillation	
Volume (mL)	**Temperature (℃)**
2.0	81.6
4.0	82.2
6.0	82.5
8.0	83.1
10	83.5
12	84.0
14	84.5
16	85.4
18	86.5
20	88.1
22	90.6
24	94.7
26	101.8


Fractional Distillation
y
28
26
24
22
20
18
16
14
12
10
8
6
4
2
Volume (mL)
80 81 82 83 84 85 86 87 88 89 90 91 92 93 94 95 96 97 98 99 100 101 102 103
x
Temperature (°C)
10 MM/CM

Discussion:

Distillation is a purification technique used to separate components of a mixture. Unlike TLC, distillation is not suitable to monitor reactions. Distillation separates the components of a mixture based on boiling points. The components have to be in liquid form or oil in order to use this technique. The higher the differences in boiling point between the components of the mixture, gives better separation. The component with the lower boiling point will be collected first the graduated cylinder in an ideal case. The boiling point is very dependent on the intermolecular forces between molecules. In this case, 2-propanol and 1-butanol both contain the same functional group called the hydroxyl group. So they both form hydrogen bonding. But since 1-butanol is one more carbon long it has the higher mass as well as more Vander-Waal and dipole-dipole forces acting on it which results in higher boiling point. Therefore, the receiving flask should be enriched in 2-propanol for the most part. In an ideal curve for of temperature vs. volume graph for simple distillation, the first point should show the boiling point of the substance with the lower BP (In our case, 82.6 degree Celsius should be the starting point roughly). Then towards the midway point of the volume, since it's a 1:1 mixture, there should be an increase in temperature all the way to the BP of the component with the higher BP (In our case that would be roughly around 117.4 degree Celsius which is the BP of 1-butanol). In this graph, the starting point is 88 degree Celsius which then drops down to 86.4 degree Celsius and then keeps on increasing in an exponential curve. There is no clear separation between the two substances. This could be due to the fact that the system lacked insulation thus cooling the 2-propanol before it got to the condenser. . Also, before we measured the first temperature the hose came off. This may have caused some evaporation. We also, observed a greasy immiscible component being collected at 113.9 degree Celsius, which I think is the 1-butanol being collected. For fractional distillation, in an ideal case the increase should be steeper in the curve. But once again, the graph looks like an exponential curve. This could be due to the fact that the insulation was done later in the procedure. The initial temperature was 81.6 degree Celsius, which is very close to the BP of 2-propanol, which is 82.6 degree Celsius. A reason for why the temperature spiked up so fast could be due to the fact that the heat produced through the transformer was too high thus causing the procedure to fasten up resulting in an inefficient separation. Better results in both techniques could be obtained with trials and more time.

Questions:

1. Explain why you must have liquid flowing back through the fractionating column in order to get a separation of the components during fractional distillation.

- Not having liquid flow back through the fractionating column results in an abnormal process of flooding. This means that if the fractionating column is too hot, then the vapour flowing up acts as an opposite force to the liquid dripping down causing a build up of this liquid in the column. The liquid gets trapped in the packing during fractional distillation. This makes distillation an inefficient process that results in a bad separation of the components of 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?

- During fractional distillation, the vapour has to rise up an added distance due to the fractionating column in a gaseous state without condensing into liquid before it reaches the condenser. The column is insulated to make sure the glass of the fractionating column towards the top is not cold enough to condense the vapour into liquid.

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

- The boiling point is reached when the pressure exerted reaches the atmospheric pressure. Therefore, the vapour pressure of benzene at its boiling point is 1atm.

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

- An increase in the atmosphere would cause the boiling point to increase, as they are directly proportional to each other. This is stated in the ideal gas law- as temperature increases, so does pressure keeping the volume constant.

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

- To make sure that the whole condenser receives water and gets cooled because if it enters from the top there is a space filled with air, which is warmer than water. Due to this you would end up having hot patches and air bubbles, which would in turn create an inconsistent distillation. This results in an uneven temperature gradient, which results in inefficient separation of the components.

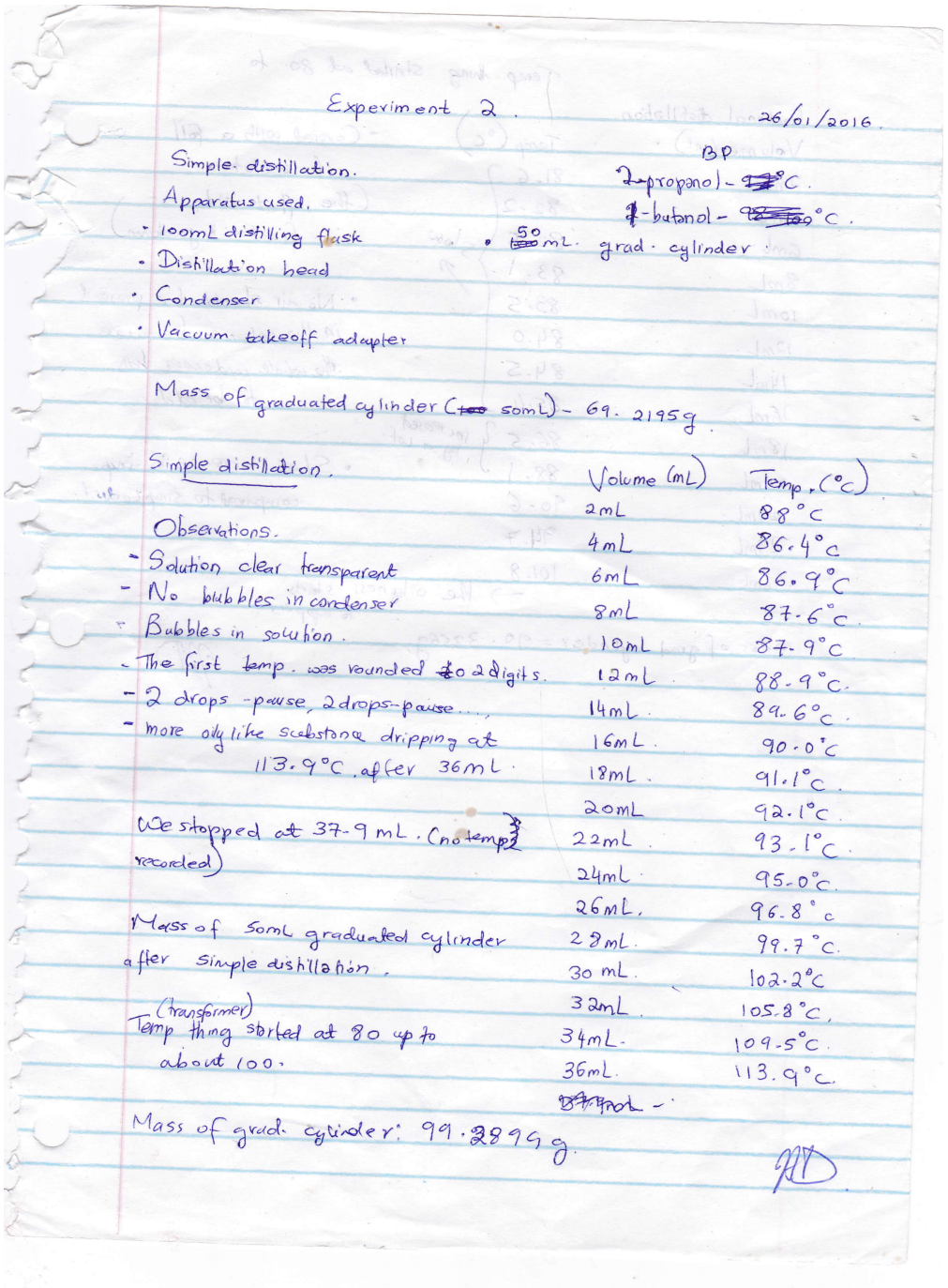
6. Compound A has a vapour pressure of 350 mm Hg at 95 °C whereas compound B has a vapour pressure of 150 mm Hg at the same temperature. If A and B are miscible, what is the vapour pressure of a 3:1 mixture of A and B at 95 °C?

- Raoult's law sates that $P_{total} = P_A \cdot N_A + P_B \cdot N_B$. In this case, A and B are in a mixture of 3:1 ratio (75% A and 25% B). This means that the mole fraction of A is 0.75 and the mole fraction of B is 0.25. Therefore:

$$\begin{aligned} P_{total} &= P_A \cdot N_A + P_B \cdot N_B \\ &= (350\text{mmHg} \cdot 0.75) + (150\text{mmHg} \cdot 0.25) \\ &= 300\text{mmHg} \end{aligned}$$

Therefore, the vapour pressure is 300mmHg.

Raw Data:



Experiment 2. 26/01/2016.

Simple distillation.

BP
2-propanol - [redacted]°C.
1-butanol - [redacted]°C.

Apparatus used.
- 100mL distilling flask
- 50mL grad. cylinder
- Distillation head
- Condenser
- Vacuum takeoff adapter

Mass of graduated cylinder (50mL) - 69.2195g.

Simple distillation.

Observations.
- Solution clear transparent
- No bubbles in condenser
- Bubbles in solution.
- The first temp. was rounded to 2 digits.
- 2 drops - pause, 2 drops - pause...
- more oily like substance dripping at 113.9°C after 36mL.

We stopped at 37.9mL. (no temp recorded)

Mass of 50mL graduated cylinder after simple distillation.

Temp thing (transformer) started at 80 up to about 100.

Mass of grad. cylinder: 99.2895g.

Volume (mL)	Temp. (°C)
2mL	88°C
4mL	86.4°C
6mL	86.9°C
8mL	87.6°C
10mL	87.9°C
12mL	88.9°C
14mL	89.6°C
16mL	90.0°C
18mL	91.1°C
20mL	92.1°C
22mL	93.1°C
24mL	95.0°C
26mL	96.8°C
28mL	99.7°C
30mL	102.2°C
32mL	105.8°C
34mL	109.5°C
36mL	113.9°C

Temp. thing started at 80 to

Fractional distillation.

Volume (mL)	Temp (°C)	
2mL	81.6	slow ↗
4mL	82.2	
6mL	82.5	
8mL	83.1	
10mL	83.5	
12mL	84.0	
14mL	84.5	
16mL	85.4	
18mL	86.5	increased by a lot
20mL	88.1	
22mL	90.6	
24mL	94.7	
26mL	101.8	→ the oilyness starts to appear.

- Covered with a foil for insulation. (the flask and fractionating column)
- No air should be present in the column because the whole condenser has to be cold.
- Slow increase in temp. compared to simple dist.

mass of grad cylinder = 90.3356g.

JD