Feb. 28th, 2014

Purifying Chemicals by Distillation

Intro:

Background

Simple Distillation:

A method of separating the components of a mixture based on the individual volatilities (boiling points) of the different components. During the beginning of a simple distillation, the lower boiling temperature liquid will phase shift into a vapour first, rise up the tube and phase shift back to liquid as it moves through the condenser and drips into the receiving flask. As such, the mixture within the receiving flask will be enriched in this liquid. As the distillation progresses and the temperature increases, more of the higher boiling point liquid will begin to vaporize, and much like like the lower boiling point liquid will make its was through the condenser tube and, now in liquid form, drip down the slanted tube into the receiving flask. The mixture in the receiving flask will now begin to become enriched in this higher booing point liquid. In practise, a mixture of two or more liquids can be collected in the same receiving flask, or, the receiving flask can be changed at the right time as to collect different distillates in different flasks.

It is important to note that that simple distillation is a physical separation process, and not a chemical reaction. At minimum, a simple distillation apparatus would include the following: A thermometer, a source of heat, distilling flask, receiving flask, distillation head, condenser tube, and a vacuum takeoff adaptor (or “elbow”).

Fractional Distillation:

A fractional distillation is similar to a simple distillation, with the addition of including a fractional column. Typically, this fractional column is “packed” with any of glass rings, metal sponge or glass wool. The concept, is that the fractional tube will induce a series of condensations and vaporization's, which effectively increase the amount of the lower boiling point liquid in the vapour thats rising up the column, at each successive mini-distillation. These mini-distillation, are referred to as *theoretical plates*. A portion of the higher boiling point liquid condenses on the material in the fractional column at each theoretical plate, and will end up dripping back down the column into the distillation flask. It stands to reason then, that once the vapour reaches the condenser tube, it is almost entirely composed of the low billing point liquid, and furthermore, the mixture in the receiving flask will be primarily of the lower boiling point mixture.

Again, as a special type of distillation, the process is a physical separation rather than a chemical reaction. At minimum, a fractional distillation apparatus would

1.*A to Z of Thermodynamics* by Pierre Perrot. ISBN 0-19-856556-9 include all items mentioned above for a simple distillation with the addition of a fractional column.

Raoult's law:

The partial vapour pressure of each component of an ideal mixture of liquids is equal to the vapour pressure of the pure component multiplied by its mole fraction in the mixture. The total vapor pressure of the ideal solution is then directly dependent on the vapour pressure of each chemical component and the mole fraction of the component present in the solution.[1]

The equation pertaining to Raoult's law is as follows:

; Where:

P_a is the partial pressure of liquid A
P_b is the partial pressure of liquid B
N_a is the mole fraction of liquid A
N_b is the mol fraction of liquid B

$$P_a = (P_a)*(N_a)$$
$$P_b = (P_b)(N_b)$$
$$P_{total} = P_a + P_b$$
$$P_{total}= (P_a)*(N_a) + (P_b)(N_b)$$

Procedure:

Simple Distillation

1. A simple distillation apparatus was assembled according to the lab manual.
2. 50.0mL of a 50:50 mixture of 2-propanol and 1-butanol were added to a distillation flask
3. The magnetic stirrer and heating mantle were turned on, and the distillation was allowed to run until approximately 48mL of distillate were collected.

Fractional Distillation

1. A fractional distillation apparatus was assembled according to the lab manual.
2. Steps 2-3 from *Simple Distillation* procedure were repeated.

Observations:

Graphs and Tables :

See attached work.

Table 1 - Quantitative observations across both distillation experiments.

Variable Property	Simple Distillation	Fractional Distillation
Electric Potential Difference of Alternator (V)	90	90
Speed setting magnetic stir (1-10)	10 initially, then 3	3
Distillation collection rate (drops/second)	dy/dt = 20 drops/29.25/s = 0.68/s	dy/dt = 50 drops/ 37.48s = 1.13/s

Qualitative Observations:

In both distillations liquids were vaporized as heat was added. This caused some of these vapours to rise into the distillation head and flow into the condenser. Once in the condenser these vapours were cooled and the resulting liquids were collected in receiving flasks.

It was visible however, that not all of the vapours made it to the top of the distilling flask. Often, liquid drops could be seen dripping back down into the distilling flask along the side of the distilling tube.

Discussion:

Discussion Questions

1. In a successful fractional distillation, a proportion of the higher boiling point liquid (in gas form) will condense at each theoretical plate and drip down the fractional column in liquid form. This is crucial in order to separate the components, as each time a portion of the higher boiling point liquid drips back down the column, the composition of the lower boiling point liquid, in the vapour rising up the column, increases. This effectively decreases the boiling point at each successive theoretical plate, allowing the vapour to move higher and higher up the column, while also increasing in composition of the lower boiling point liquid. By the time it reaches the junction between the fractional column and the condenser tube, it will be almost entirely composed of the lower boiling point liquid, at which point it will move through the condenser tube, chaining in phase to liquid, and drip into the receiving flask. If the distillation was successful, the receiving flask mixture will be almost entirely composed of the lower boiling point liquid while the distillation flask the distillation flask will mixture will be almost entirely composed of the higher boiling temperature liquid.
2. A uniform temperature gradient in the fractional column means that the temperature at each theoretical plate will be different. This is important if the distillation is to run successfully, as a fractional distillation depends on the fact that ….. FINISH
3. The boiling point of any substance, by definition, is the temperature at which the vapour pressure of the substance is equal to the atmospheric pressure. Assuming the atmospheric pressure is 101.325KPa or 760 Torr, the vapour pressure of benzene at 80 degrees celsius must also equal 101.325KPa or 760 Torr.
4. At lower pressures, liquids boil at lower temperatures. Thus, a liquid subject to atmospheric pressure will boil at a *lower temperature* if the pressure is *lowered below* that of atmospheric pressure. A liquid will boil at a *greater temperature* if the pressure is *increased above* that of atmospheric pressure.
5. It is important to have water enter the bottom of the condensing column in order to ensure that the water jacket remains full with water. If the water was to enter the top of the condensing column, it would flow to quickly down the slope of the column, and because of this reason the water jacket would never be completely full. It is important that the water jacket is completely full in order to ensure maximum heat transfer as the vapours move through the condenser. When water enters the column through the bottom, it must fight gravity to move up the slope - this sets up a steady flow within the water jacket that results in it completely filling with water.

6.

Where;
Na = ¾
Nb = ¼
Pa = 350 mmHg
Pb =150mmHg
Pt = ? (mmHg)

$$
\begin{aligned}
P_{total} &= (P_a)*(N_a) + (P_b)(N_b) \\
&= (350\text{mmHg})*(¾) + (150\text{mmHg})*(¼) \\
&= 300\text{mmHg}
\end{aligned}
$$

Therefore the vapour pressure of a 3::1 mixture of A and B at 95^0C is 300mmHg

General Discussion**:**

During a simple distillation involving two compounds, the more volatile compound (lower bolding point) will begin to vaporize first and move up the column, condensing as it moves through the condenser tube and collects in the receiving flask. When temperature vs volume of distillate is graphed, the collecting of the more volatile compound corresponds to an initial increasing slope. At the point where approximately all of the more volatile compound has been collected, there will be a point of inflection on the aforementioned graph. Directly after this point, the temperature will have increased enough so as to vaporize the less volatile compound (higher boiling point). This compound will rise though the column collect in the receiving flask as it moves through the condenser tube and condenser. This will correspond to a decreasing curve on the graph, as the rate of collection of the more volatile compound decreases as the amount of it left in the distilling flask decreases. The entire curve eventually flattens out, marking the point where all of the original mixture has been collected in the receiving flask.

Our results, in graphical form, do resemble the expected graphical output from a simple distillation. There is a clear distinguishable increasing slope as the temperature begins to increase, a point of inflection marking the complete evaporation of the more volatile compound, and a decreasing slope directly after representing the period in which the less volatile compound begins to become evaporate and condense into the revving flask. The entire curve also eventually flattens out - marking the point where all of the original mixture has been collected.

In a distillation involving the use of a fractionating column, a series of condensations and vaporizations (mini-distillations) occurs enriching the vapour in the substance with the lower boiling point. Since the density of the packing, the surface area of the packing material, and the surface area of the inside of the column all effectively improve the separation of the components of the mixture during distillation as they increase, we expect to observe two distinct temperatures at which distillates are being collected at, corresponding to each substance’s boiling point temperature.

The plot of these temperatures as a function of time is expected to resemble a logistic curve which is very flat at both ends (≈ constant temperatures) and rises sharply at the point in time where all of the substance with the lower boiling point has been removed through distillation and the substance with the higher boiling point is now being distilled. Although we did not collect data in reference to time as an independent variable during this experiment, we can say that our temperature vs. volume of distillate collected curve should have the same general shape as the expected temperature vs. time graph. This is because the collection rate was intentionally kept as constant as possible, meaning that a plot of volume of distillate collected vs. time would demonstrate a linear relationship. When superimposing a curved function and a linear function, the curve retains its shape and generally just alters its path to follow the general path of the linear function.

However with this being said our experimental data did not support this hypothesis. The graphical representation of collected data from this experiment showed a scattered array of temperatures and did not follow the logistic model. Furthermore, the recorded boiling point temperatures dropped significantly after 18 mL of distillate were collected, which is evidently wrong since the second substance being distilled inherently must have a higher boiling point temperature than the first. Although, unknown at the time, what appears to have occurred is a flood in the fractionating column. We believe this occurred because the volume flow rate of the distillate was far too great, and in comparing quantitative values collected during both distillation we can see that it was in fact much higher during the fractional distillation than it was during the simple distillation (see **figure 1.**).

This mistake was not corrected *during* the lab simply because we were not aware of the mistake we were making. Careful review of the theory behind fractional distillations has allowed us to understand our mistake *after* the performance of the lab and to make this hypothesis as to what, in fact, caused the flooding of the fractional column.

Conclusion: Quantitatively and graphically, we were able to demonstrate simple and fractional distillations, both on an initial mixture of two compounds with varying boiling temperature. While our results (graphically) agree quite strongly with the expected results as they pertain to simple distillaion, that do no agree with the expected results (graphically) as they pertain to fractional distillation. The cause of this, was determined to be an experimenter error.

Table 2 · Simple Distilation

Temperature (^{0}C)	Volume of Distillate (mL)
86	2
88	4
89	6
90	8
91	10
91	12
92	14
93	16
94	18
95	20
96	22
98	24
99	26
101	28
103	30
105	32
108	34
110	36
112	38
114	40
115	42
116	44
117	46
117	48

Graph 1. - Temperature of Distillation Vs. Volume of Distillate collected (simple distillation)

2

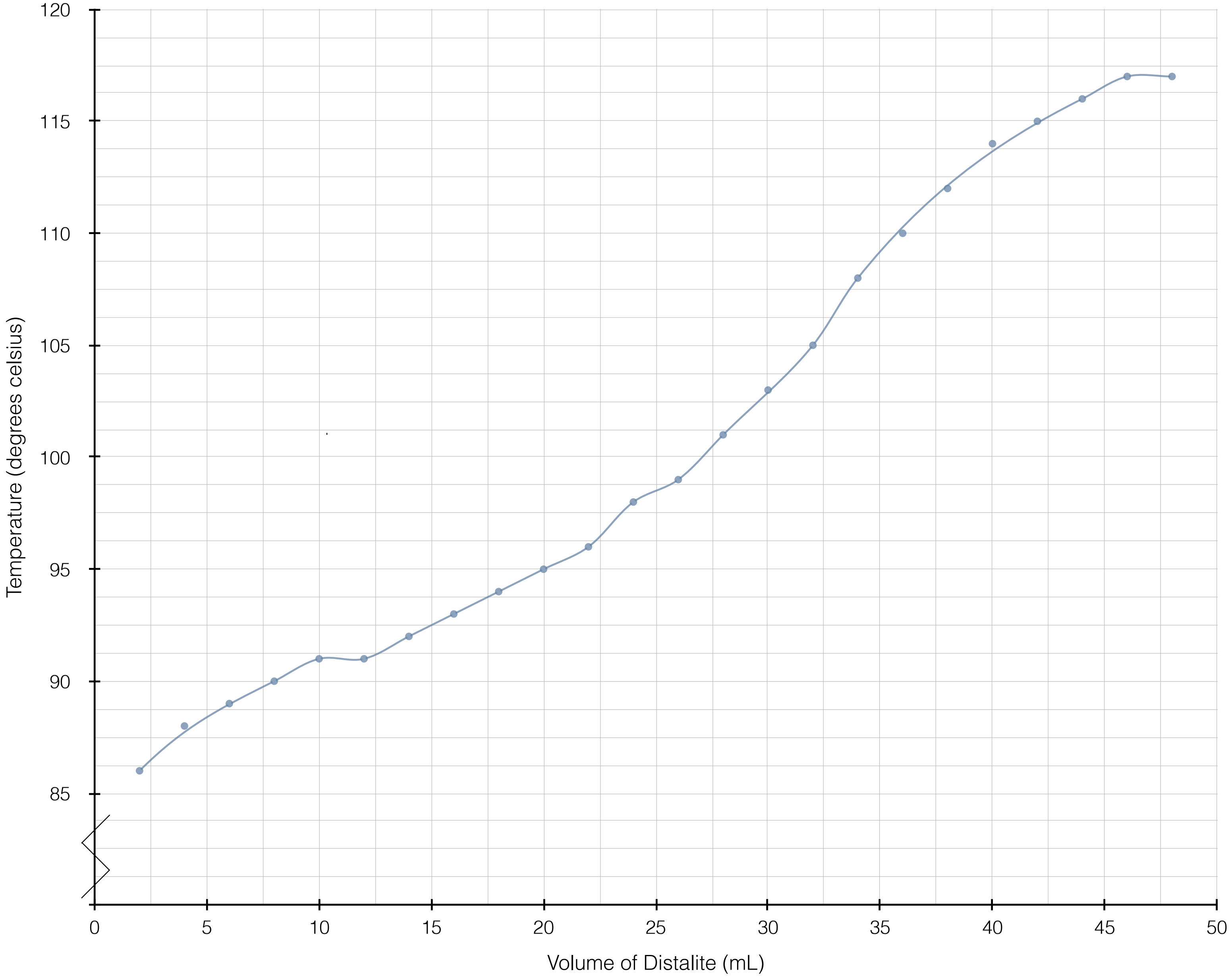

Table 3 · Fractional Distilation

Temperature (ºC)	Volume of Distillate (mL)
78	2
82	4
83	6
84	8
85	10
85	12
86	14
87	16
87	18
83	20
77	22
72	24
67	26
63	28
65	30
68	32
70	34
73	36
74	38
73	40
73	42
68	44
68	46
58	48

Graph 2. - Temperature of distillation Vs Volume of Distilate Collected (fractional)