Distillation and fraction distillation lab report assignment

By: Sean Polson 05 September 2011 Organic Chemistry Lab: Experiment 1 Section 2 ??? 12: 30 to 2: 20 Distillation and Fractional Distillation Distillation and Fraction Distillations Purpose: The purpose of distillation is to purify a liquid. Distillations are use to purify contaminates out of water to obtain clean pure water, as well as, to separate mixtures of liquids into their individual components; e. g. methanol and water. Objective: Distill methanol from water using a simple distillation apparatus and fractional distillation apparatus to determine which is a more accurate form of distillation. Chemical reaction and mechanism: N/A

Procedure: This experiment is conducted in three phases: 1) Phase I: Simple Distillation of a Pure Compound 2) Phase II: Simple Distillation of a Mixture 3) Phase III: Fractional Distillation of a Mixture Phase I: Simple Distillation of a Pure Compound Distill a 15 mL sample of methanol in a 50 mL boiling flask using a simple distillation apparatus. Ensure a boiling stone was added to the boiling flask before attaching to the distillation apparatus. Additionally before the distillation began, all pieces of the apparatus were secured with ring stands and clamps and the heating mantle was able to be adjusted to prevent heating to fast.

The temperature of the sample was recorded when the first drop of distilled solution was collected and every mL afterwards. Heating was stopped before the 15 mL sample dries to prevent shattering of the boiling flask. Additionally each mL of distilled solution was weighed and the refractive index was identified. Phase II: Simple Distillation of a Mixture Set up a simple distillation apparatus as in Phase I. In a 50 mL boiling flask, 7 mL of methanol and 7 mL

of water, as well as, a boiling stone were added. The boiling flask was attached to the apparatus and heating was started.

The temperature was recorded when the first drop of distilled solution was collected and every mL afterwards. The distilled solutions were weighed and the refractive index was identified. Phase III: Fractional Distillation of a Mixture Set up a fractional distillation apparatus. In a 50 mL boiling flask, 15 mL of methanol, 15 mL of water, and a boiling chip were added. The boiling flask was attached to the apparatus and heating was started. The temperature was recorded when the first drop of distilled solution was collected and every mL afterwards until a temperature of 100oC was reached and 1-2 mL of solution was collected afterwards.

Additionally each mL was weighed and the refractive index was identified. Data: Phase I: The initial temperature and boiling point temperature were collected. Additionally the temperature, weight, and refractive index were collected for each mL collected. Initial Temperature: 23oC Boiling Point Temperature: 26. 5oC mL Solution Collected| Temperature (oC)| Weight (g)| Refractive Index (nD)| 0| 23. 0| -| -| 1| 60. 0| 2. 10| 1. 327| 2| 61. 0| 2. 02| 1. 326| 3| 62. 0| 1. 33| 1. 199| 4| 62. 0| 1. 33| 1. 199| 5| 62. 0| 1. 33| 1. 199| 6| 62. 0| 1. 33| 1. 199| Table 1. Phase II: The initial temperature, boiling point temperature, and 1st drop temperature were collected. Additionally the temperature, weight, and refractive index were collected for each mL collected. Initial Temperature: 31oC Boiling Point Temperature: 33oC 1st Drop Temperature: 65oC mL Solution Collected| Temperature (oC)| Weight (g)| Refractive Index (nD)| 0| 23. 0| -| -| 1| 70. 0| 2. 18| 1. 312| 2| 74. 0| 1. 42|

1. 337| 3| 65. 0| 1. 04| 1. 335| Table 1. 2 Phase III: The initial temperature, boiling point temperature, and 1st drop temperature were collected.

Additionally the temperature, weight, and refractive index were collected for each mL collected. Initial Temperature: 23oC Boiling Point Temperature: 25oC 1st Drop Temperature: 62oC mL Solution Collected| Temperature (oC)| Weight (g)| Refractive Index(nD)| 0| 23. 0| -| -| 1| 64. 0| 0. 608| 1. 338| 2| 64. 0| 0. 574| 1. 338| 3| 64. 0| 0. 496| 1. 332| 4| 64. 0| 0. 454| 1. 332| 5| 64. 0| 0. 490| 1. 333| 6| 66. 0| 0. 951| 1. 333| 7| 85. 0| 0. 475| 1. 333| 8| 93. 0| 1. 014| 1. 341| 9| 96. 0| 0. 347| 1. 342| 10| 96. 0| 1. 466| -| 11| 97. 0| 0. 588| 1. 335| 12| 97. 0| 0. 21| 1. 333| Table 1. 3 Actual Weight (Density) of 1mL Methanol: 0. 791 g/mL http://students. chem. tue. nl/ana21/Safety/Chemical %20Properties. htm Actual Refractive Index of 1mL Methanol: 1. 326 http://www. sigmaaldrich. com/catalog/ProductDetail. do? D7= 0&N5= SEARCH_CONCAT_PNO%7CBRAND_KEY&N4= 269824%7CALDRICH&N25= 0&QS= ON&F= SPEC Discussion: When the solution is heated in the boiling flask, it evaporates into its vapor form and travels up the distilling apparatus. As it reaches the distilling side arm, the temperature of the vapor is collected.

The vapor pressure becomes strong enough that the vapor begins to travel down the condensing tube where it is converted back into liquid. This liquid should be distilled from any contaminants. This is capable because different molecules have different boiling points. For instance waters boiling point is roughly 100oC where as Methanol’s boiling point is 65. 4oC therefore the Methanol will boil and evaporator first leaving only water in the boiling flask. Once the temperature of the vapor reaches 100oC no methanol should be

left in the boiling flask and the water should now be the only vapor that is being collected.

In Phase I, after initial setup of a simple distillation apparatus, the initial temperature was collected in order to have a base line temperature before the experiment/distillation began. Additionally the boiling point temperature was captured at the point when the solution in the boiling flask first began to boil. This temperature was gathered in hopes to compare with the known and documented boiling points of methanol and water in chemistry encyclopedias. However, this temperature was found useless as the location f the thermometer was at the top of the distillation apparatus and the boiling occurred at the bottom of the apparatus, thus the boiling point temperature gathered was inaccurate. The temperature that was being gathered was the vapor temperature once the vapor finally reached the top of the apparatus. A 25 mL graduated cylinder was used to collect the 1 mL sample of the distilled solution as it dropped from the distillation apparatus. Due to lack of 10 mL graduated cylinders, a potential error could be that too much or too little solution was gathered; not an exact 1 mL sample was collected due to size of cylinder.

This solution was then weighed and ran through the refractive index machine. An additional graduated cylinder was used to collect the next 1 mL sample while the 1st sample was being weighed and refractive index collected. To weigh the 1 mL sample, a small beaker was set in a zero gravity scale to be calibrated to zero, once calibrated, the 1 mL sample was transferred from the graduated cylinder to the beaker and weighed. A potential error in this weight would be that not every drop made it out of the

cylinder before weighing therefore providing a less than accurate measurement.

The weights and refractive indexes were recorded in table 1. 1. Additionally the actual weight and refractive index of 1mL solution of Methanol was collected from an internet encyclopedic source, in order to compare the accuracy of a simple distillation apparatus. Our final weight of 1. 333g/mL is 0. 542 g/mL heavier than the 0. 791 g/mL weight found on the internet source, therefore showing that our distilled solution was not 100% distilled of all contaminants and needs to be further distilled. Additionally the refractive index of our distilled sample was 1. 99 and the actual refractive index of methanol is 1. 326. Our results reflect an additional error in collection. Our final three mL of sample was collected together rather than in 1 mL increments therefore our weights and refractive index are averages of all three. A possible method of collection that could be used is to use a graduated cylinder to collect 1 mL samples, once each mL is collected, immediately transfer to a small, labeled, beaker and set aside until end of collection before weighing. This will ensure enough time is allotted for the accurate gathering of 1 mL samples.

This method is used in phase III. Phase II of the experiment was set up exactly the same as phase I, however water was mixed with the methanol solution in the boiling flask. The purpose of this phase of the experiment is to separate the two solutions using different boiling temperatures in a simple distillation apparatus. Again the initial temperature was gathered to have a base line temperature before beginning the distillation. Also the boiling point temperature was gather, however later determined it was not necessary.

The First drop temperature was recorded as well. This temperature reading is used to determine which solution is being collected. If the temperature read roughly 65oC the solution being gathered is Methanol, however if the temperature read roughly 100oC the solution being gathered would be water. This temperature is important as it gives us a rough idea of what we are collecting. From our data under Phase II, we know that the solution initially being gathered was Methanol since the temperature of the first drop was roughly 65oC.

Again the solution was gathered in a 25 mL graduated cylinder and the weight and refractive index was collected for each mL collected and recorded in table 1. 2. Like Phase I after each mL sample was collected, an additional 25 mL graduated cylinder was used to collect the next mL sample as the first sample was being weighed and refractive index recorded. The same errors could be found in Phase II as where found in Phase I due to the size of the graduated cylinder and transfer from cylinder to beaker. Our results of 1. 04 g/mL are 0. 249 g/mL heavier than the actual 0. 91 g/mL weight of methanol, therefore showing that our sample is not 100% clear of water and will need further distillation before being properly distilled. Additionlly the 1. 335 refractive index gathered is 0. 009 higher than the actual 1. 326 refractive index for methanol, further showing that our distillation is not 100% distilled. Phase III of the experiment was set up like Phase I and II however a fraction column was connected to the boiling flask. The purpose of the fraction column is to cause the rising vapor to cool and condense and then evaporate ultiple times as it travels up the fraction column before finally reaching the side arm of the distillation apparatus. This

causes the mixed solution to actually distill multiple times before being collected. From our results in phases I and II this extra tool is beneficial as we found that our distillation needs to be distilled more than once to be completely free of any contaminants. Like phases I and II the initial temperature was gather for a baseline temperature and the boiling point temperature was gathered (again not needed). Furthermore the temperature of the first drop was recorded.

The importants of this was described above. From our results we know that Methanol is being collected first since the first drop temperature was 62oC. Once the first mL sample was collected, we immediately transferred it to a small beaker labeled “ 1” and set off to the side for weight and refractive index measurements to be collected at the end of the distillation process. Again we utilized a second graduated cylinder to collected samples as our first graduated cylinder was being emptied and cleaned (25 mL graduated cylinders were used).

This was done to prevent the gathering of more than 1 mL sample at a time. Roughly halfway through the distillation, the temperature jumped into the 90oC range indicating that all methanol solution was burned off and collected and the samples being collected now were water. From our results our methanol was done being collected after the 6th mL according to table 1. 3. Sample 7 (7th mL) in table 1. 3 would have some methanol, however it would be mostly water as the temperature had rose from 66oC to 85oC. The weight of sample 6 (6th mL) was 0. 951g/mL which was only 0. 16 g/mL higher than the actual 0. 91 g/mL indicating that a fractional distillation is better than a simple distillation, however futher distillation would still be

needed to get a 100% measurement. Again transfer of sample from graduated cylinder to beaker could be a potential error in measurement. Our refractive index tells the same thing as it is 0. 009 higher than the actual refractive index of methanol. The lack of refractive index measurement in sample 10 (10th mL sample) indicates another potential error in both weight measurement and refractive index measurement. The lack of a sample value is due to evaporation of the sample as it sat in the beaker to be measure.

Since this occurred with sample 10 it had to have occurred with all 12 samples as a result our weights and refractive index values could be slightly squid. A way to fix this problem for future experiments would be to cover the sample in the beaker with a wax film to prevent any evaporation to occur. Conclusion: From the results gathered in the three phases, a fractional distillation apparatus is more effective and time efficient than a simple distillation apparatus, because it distills the solution multiple times before finally being collected.

The simple distillation did not result in an efficient separation of methanol and water, however the fractional distillation did. In the Simple distillation, there was a 0. 249 g/mL higher difference than the actual weight of methanol where as the fractional distillation only had a 0. 16 g/mL higher difference, resulting in a better distillation. The fractional distillation could be improved with better insulation of the fraction column to allow the distillation process to occur more efficiently. Additionally heating the solution in the boiling flask slower would allow for a more efficient separation of methanol and water.