CHEM 2219: Exp. #5 Simple Distillation

Objective: In this experiment you will learn to separate the components of a solution using simple distillation methods; and, identify the liquid component by the boiling point and refractive index determination and the solid by the melting point determination.*

* The boiling point and refractive index (RI) are two useful physical properties of a liquid. They are used for identification purposes. The RI is additionally used as a measure of purity of the sample being examined. The melting point (MP) is a physical property of a solid also used for the purpose of identification and purity determination.

Reading Assignment: MTOL, pp. 72-85 (distillation theory), 49-52 (refractive index), 52-56 (melting point): OCLT, pp. 44-47 (heating & cooling methods), 249-252 (overview of distillation), 276 (microscale distillation), 309-315 (melting point).
Also on **CANVAS** read and answer prelab questions for – **Simple Distillation**

Concepts:
Boiling Point, Condensate, Condensation, Distillate, Distillation, Evaporation, Melting Point, Reflux, Refractive Index, Theoretical Plates, Vaporization

Chemicals:
Liquids: 2-Butanone, Ethyl Acetate, Toluene
Solids: Fluorene, 2-Nitroaniline, 3-Nitroaniline

Safety Precautions:
Wear chemical splash-proof goggles and appropriate attire at all times.
2-Butanone, ethyl acetate and toluene are flammable liquids.
Fluorene, 2-nitroaniline and 3-nitroaniline are combustible solids.
Hot glassware looks just like cold glassware. Hot aluminum looks just like cold aluminum.
Be careful when working with hot glassware and hot aluminum blocks!
Do not to touch items on the hotplate!
Use crucible tongs to disassemble hot glassware and remove items from the hotplate.

Materials:
aluminum block, beaker (100ml), conical vial (5 ml), crucible tongs, disposable pipets (2) & bulb (1), disposable vials (2) and caps (2), finger clamps (2), Hickman still head, hot plate with magnetic stirrer, labels (2-small), magnetic spin vane, ringstand, Teflon septum with hole in the center, and thermometer

Instruments:
Melting Point Apparatus & Refractometer

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Background Information:

The use of distillation to separate the components in a mixture is based on the principle that the boiling liquid in the vial and the vapor produced have a different composition. When a solution of a liquid containing a dissolved solid is heated to the boiling point of the liquid, the vapor will have a higher concentration of the liquid (i.e., the more volatile component of the solution / the component with the lower boiling point). The vapor rises up the glassware where it cools and condenses. When the vapor condenses, it is called the distillate. When enough distillate has collected in the Hickman still head, it can easily be removed using a pipet. The distillate will then be enriched in the more volatile component of the solution.

Distillation can also be performed on a solution of multiple liquids. In this case, the liquid with the lowest boiling point will be the most enriched in the distillate, regardless of whether it is the major component of the solution, because it is the one that is the most volatile.

Distillation can also be utilized to manage natural resources. It is an indispensable technique for obtaining drinking water from seawater. Distillation is the oldest and still most widely used technology for desalination (removal of salt from saltwater). In the petroleum industry, oil refineries use distillation to transform crude oil into fuels and chemical feed stocks. Distillation is also employed by the alcohol and brewing industry to increase the alcohol content of fermented products.

In this experiment, simple distillation will be used to separate an organic liquid from an organic solid. Simple distillation involves a single cycle of vaporization and condensation. Simple distillation is used to purify liquids that contain either nonvolatile impurities, such as salts, or very small amounts of higher- or lower-boiling liquids. Simple distillation is not a practical method for separating compounds with similar boiling points.

In order to separate liquid mixtures where the components have similar boiling points and/or are present in comparable amounts, fractional distillation must be employed. In fractional distillation, insulated fractionating columns permit multiple cycles of vaporization and condensation in a single operation. The column consists of closely spaced packing material or "plates." The vapor condenses on multiple surfaces in the fractionating column and the resulting liquid revaporizes. At each stage in the series of vapor–liquid equilibrium, the vapor becomes more enriched in the more volatile (lower-boiling) component. Given a sufficient number of "plates," the mixture will distill in fractions. Each fraction consisting of only a single pure substance.

In the next experiment, two organic liquids will be separated from each other using fractional distillation. However, since the number of plates will be low, the distillate will simply be enriched in the component with the lower boiling point, rather than a pure substance.

References:

Distillation. Wikipedia article. Available January 15, 2021 at: https://en.wikipedia.org/wiki/Distillation

Simple Distillation. Flinn Scientific. Available on October 4, 2020:
https://www.flinnsci.com/api/library/Download/224aed85de4d4fa287d0eceef9a40adc

Simple Distillation Procedure

You are provided with a 2 component solution: an organic solid of low volatility in a solvent which can be distilled readily at a temperature not higher than 120 °C. Your task is to separate the components by distillation, record the boiling point (range) for the distillate and its refractive index, record the melting point (range) for the remaining solid, calculate the % error (by comparison with literature values) for MP, BP & RI in the recorded observations, and in your conclusion comment on any similarities or discrepancies.

1. Preheat the hot plate and Al block at a heat setting of ~130-145 °C while you assemble your glassware.

2. Put together a simple distillation set-up.*
(See Figure 1. on the right.)

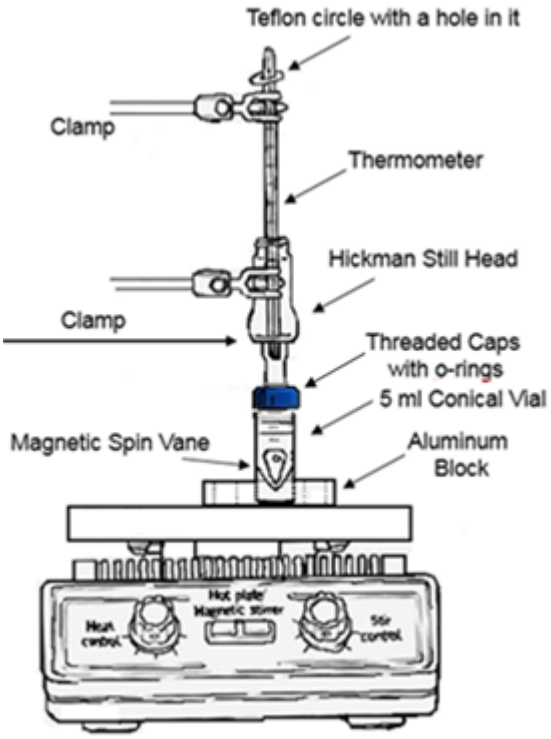

Figure 1: Distillation Set-up.

***Note:** Verify that there is an o-ring below the cap of the Hickman still head before attaching the conical vial to the Hickman still head. If there is a sidearm on the Hickman still, verify the arm is capped and that the cap has a septum in it. **Do <u>not</u> lower empty glassware into the Al block; it will cause the glassware to crack.** Once the unknown has been added to the conical vial, it may be lowered into the Al block. You do <u>not</u> have to wait for the temperature to get to 130 °C before lowering the glassware. **Do <u>not</u> worry about adjusting the thermometer height until the glassware has been lowered into the Al block.**

3. Acquire an unknown from the hood. Record the unknown number and physical properties of the unknown in lab report.

4. Transfer 3.0-3.5 ml of the unknown solution to a 5-ml conical vial. Record the actual volume.

5. Add the 5/8" magnetic spin vane (point down V) to the vial. (The spin vane will help regulate boiling and avoid bumping.) Attach the Hickman still head and thermometer as shown.*

***Note:** Be sure to add a Teflon septum with a hole in it around the thermometer. The septa goes above the clamp to keep the thermometer from sliding into the vial and breaking. **Do <u>not</u> clamp the thermometer too tightly as it may cause the thermometer to break.** Make sure the clamp is tight enough though that the thermometer does not tilt and fall over. The bulb of the thermometer should be positioned so that the bulb is completely in the neck of the Hickman still head. The top of the bulb should be at the top of the neck.

6. Adjust the temperature of the hotplate in order to regulate the boiling rate so that a smooth slow distillation is attained.

7. Acquire from the supply cart, 2 disposable vials with caps, 2 small labels, 2 disposable pipets and 1 pipet bulb. Label one vial **(F)** for FORERUN and the other **(MF)** for MAJOR FRACTION. Place the vials in a 100 ml beaker so they stand upright and do not tip over.

Note: If you have not used snap cap vials before, you might practice putting on and removing the caps before adding chemicals to them. The caps "snap" on the vials – place the cap over the vial and press down. The easiest way to remove the cap is to run your thumb up the side of the vial and push the cap off the vial.

8. Note the temperature when the distillate first starts to condense (become foggy) in the Hickman still head. Record the temperature when the condensation begins to flow down the walls of the Hickman still head. (*This is the beginning of your boiling point range*.)

9. Allow the distillate to accumulate in the Hickman still until the well is at least half full. Record the temperature when the still is approximately half full. (*This is the top of the BP range for the forerun*.)

10. Using a pipet, transfer this accumulated first fraction to the 1-mL disposable vial labeled "F" and cap it.

Note: About 1/4" of liquid in the vial is an adequate sample. Keep vials in a 50ml beaker so it can't tip over & spill. Make sure to cap the vial. If you need to wait for more liquid to accumulate so that you can add it to the sample, leave the cap on the vial until you are ready to add more of the sample.

11. Continue the distillation of the remaining solution and record the boiling point range. (*Again record the temperature when the distillate first starts to condense and flow down the walls of the Hickman still head and just prior to removal of the sample. Allow the distillate to accumulate in the Hickman still head until it is at least half full*.) Using a clean pipet, transfer this second fraction to the vial labeled "MF" and cap the vial. Keep acquiring distillate until the major fraction vial has at least 1/4" of liquid in it.

Note: There should be less than 1 ml of liquid remaining in the bottom of the conical vial. If the distillate fractions have all been collected and more liquid still needs to be removed, disassemble the glassware. Remove the thermometer. Lower the glassware to the benchtop. **Do <u>not</u> place hot glassware on metal because it may cause the glassware to crack!** Using crucible tongs, disconnect the Hickman still head from the conical vial. Place the conical vial back in the aluminum block on the hotplate. This will allow the vapors to evaporate directly. If necessary, lower the hood to reduce exposure to the chemical vapors.

Keep heating the solution until bubbles are no longer visible. **AVOID EXCESSIVE HEATING AT THIS POINT** which may cause decomposition of the remaining solid. Once the bubbling has stopped, the liquid remaining is actually melted solid that will not evaporate, but may decompose if heated excessively. This will adversely affect the MP.

12. Remove the distillation apparatus (or the conical vial) from the heat source. Keeping the apparatus clamped to the ring stand, lower the apparatus until the vial is standing on the desktop. Allow the apparatus to cool down to room temperature, whereby the solute will crystallize out.

Note: Again do <u>not</u> place the hot vial on the metal ringstand as it may cause the vial to crack.

The refractive indices for the forerun and major fraction may be determined while the solid cools. (*Skip to step 15. Then come back to step 13*.)

13. Remove the solid from the vial with a microspatula onto a piece of dry filter paper. Allow the solid to cool and dry out completely before checking its MP.

Note: The solid may have formed around the magnetic stir bar. It can easily be chipped off the stir bar with the microspatula. Hold the stir bar with your forceps and be careful when removing the solid to keep the sample on the filter paper. If sample is still damp, then some solvent may still be present. You may help quicken the drying of the solid by folding the filter paper over the sample and patting the sample with the filter paper to squeeze out any excess solvent.

14. Record the model name and number of the Melting Point apparatus used. Consult the Melting Point Determination handout for operating instructions.

15. Determine the refractive indices of the two fractions on the Abbe or digital refractometer. Record the brand/type of which refractometer you used and your results. Record the temperature of the refractometer cooling water in order to do the RI corrections for temperature. Consult the Refractive Index handout for operating directions.

16. Dispose of chemicals in the appropriate containers. Clean* and return all glassware and equipment to their original locations.

***Note:** In order to maintain social distancing and reduce congestion at the waste hoods, the recommended method for cleaning the glassware is to rinse the used glassware items with acetone into a waste beaker. For this reason, a bottle of acetone, cotton swabs and Kim wipes have been placed near the balance. Once the items have been cleaned and put away, the contents of the waste beaker can be poured into the waste container in the hood, cleaned with acetone and returned to the desk.

Post Lab:

1. Compare your experimental values for the BP and corrected RI to those in your prelab table in order to identify the liquid in your unknown and the MP to identify the solid.

2. Show calculations for Refractive Index corrections. The Refractive Index that is reported as nD_{20} in tables corresponds to the refractive index of the material at 20°C. So in order to compare your answer to the value in the table you must first "correct" it using the equation provided in the handout and shown below.

$$nD_{20} = nD_{observed} + 0.00045 \text{ (Temperature observed} - 20\ ^{o}C)$$

3. Show calculations for at least one of the Percent Error equations for your reported MP, BP and corrected RI.

$$\% \text{ Error} = [(\text{theoretical} - \text{observed}) / \text{theoretical}] \times 100$$

4. Tabulate the literature values, actual values and % errors observations.

5. Discuss how the identification of the liquid component and the solid component were made.
   For example, explain which physical properties were used to determine each component.

6. Discuss how successful the experiment was and make suggestions for how to improve the experiment.

Examples include, but are not limited to:

1. If the two components were separated and identified, the experiment was successful.

   Suggestions might be made for how to make the procedure more efficient.

2. On the other hand, if the melting point could not be readily determined because the solute did *not* solidify due to residual solvent and had to be determined the following week, then the experiment was not as successful as it could have been.

   Suggestions might be made for how to more effectively remove any excess solvent.