ORGANIC CHEMISTRY LABORATORY

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2011
Melting point

1. What two effects do impurities have on the melting point of an organic compound?

Soluble impurities affect the melting point of a solid in the following manner.

a. The presence of an impurity in the molten compound reduces its vapor pressure thus lowering the melting point of the compound.

b. Broaden the melting point range.

2. For what two purpose are melting points routinely used

a. To determine the identity of an organic solid.

b. To determine the purity of an organic solid.

3. What effects on the measured melting point would you expect in each of the following cases?

   a) Presence of pieces of glass in the sample
      No effect.

   b) Presence of solvent within the crystals
      Melting point depression, Melting point range broaden.
      Sample must be dry because moisture from solvents like water and other solvents will depress the melting point like an impurity.

   c) Using too large sample when determining the melting point
Too much material will broaden range & may increase melting point

**Lab Questions**

1. **Define the following**
   A. Melting point
   B. Intermolecular forces
   C. Hydrogen bonding

2. **Arrange the following IMF according to their strength**
   Dipole-Dipole interaction forces, Hydrogen bonding, Van der Waals forces

3. **How does the atmospheric pressure affect the melting point?**

4. **What is the purpose of determining the melting point in this lab?**

5. **Why is this method not used for finding the melting points of inorganic ionic compounds?**

**Recrystallization Questions**

1. **What is the purpose of recrystallization?**
   It is the primary method of purifying crude solid compounds.

2. **What is the recrystallization method based on?**

   It is a process based on the differences in solubilities of solids in a given solvent system.

3. **How can you increase the solubility of a compound?**
   You can increase it with added heat.

4. **How do you get rid of insoluble impurities?**
   They are removed through the method of hot filtration.

5. **How do you separate the purified solid crystals from the surrounding liq. (called Mother Liquor)?**
   Suction or Vacuum Filtration.
6. What should you do if the single solvent does not fulfill all the four requirements?
You should create a "Solvent Pair" where a second solvent miscible with the first solvent is added.

7. How should the "solvent pair" work?
One solvent should dissolve the solute well while the other should dissolve the solute poorly.

8. When must a Hot Filtration be done during a recrystallization?
There are insoluble impurities remaining in the solution and there are colored, soluble impurities which necessitates charcoal.

9. Why are the apparatus used in the Hot filtration process heated before use?
To prevent pre-mature precipitation of the solid on the funnel or in the receiver.

10. What is Charcoal used for?
They are used to remove colored, soluble impurities

11. How does Charcoal work?
The colored substances attach to the activated surfaces of the Charcoal by adsorption.

12. At what temp. are the best crystals obtained?
At room temperature

13. What is a disadvantage of using Ice temp. for recrystallization?
If the flask is cooled in an ice bath crystallization time is shortened but crystal size may be smaller; higher yield.

14. What are some other methods to initiate crystal growth?
Scratching, seeding, and reducing the volume of the solvent through evaporation or adding and incompatible solvent to the sys.

**Answers for Recrystallization Experiment’s questions**

1. **Why is it recommended to use no more than the minimum amount of solvent than is necessary to dissolve the solid being recrystallized?**

   A minimum amount of solvent will minimize the amount of desired compound that remains dissolved in the solvent and must be thrown away.

2. **Why should the hot filtrate be allowed to cool slowly as the desired product recrystallizes?**

   A cooling rate that is too rapid is likely to entrain or trap impurities in the crystals being formed. Also slow crystallization promotes the formation of larger crystals which easier to handle and wash.

3. **For what purpose is charcoal used in recrystallization?**

   To removed colored impurities by adsorption.

4. **How are insoluble impurities removed during recrystallization?**

   Removed by hot gravity filtration.

5. **How are soluble and insoluble impurities removed during recrystallization?**

   Soluble impurities removed by suction filtration after cooling.

   Insoluble impurities removed by hot gravity filtration.

6. **Why must the flask and funnel be heated before the hot solution is filtrated?**

   To prevent pre-mature precipitation of the solid on the funnel or in the receiver.
7. Why fluted filter paper used for a hot filtration?

Allow the liquid to pass through it very quickly and give you a lot of surface area on which collect the solid “impurities”

8. Mention three properties a solvent should have to be suitable for recrystallization a particular organic compound?

1. purified compound must be very soluble in hot solvent and almost insoluble in cold solvent.

2. It dissolves the impurities completely at room temperature.

3. The solvent must not react with substance to be purified.

4. Cheap, non-flammable and non toxic .

5. Volatile solvent (Low Boiling point). easily removed

6. It should have boiling point lower than the melting point of the solid to be purified.

Extraction

1. What are the properties of a suitable solvent for the extraction of an organic solute from an aqueous solution?

   1. Immiscible with first solvent.
   2. Solubility of extracted solute in it is high.
   3. Non flammable, non toxic and cheap.
   4. (Easily removed from solute after extraction.)

2. Why should the stopper be removed from the separatory funnel before liquid can be withdrawn through the stopcock?

To balance the pressure in the separatory funnel with atmospheric pressure.

لاّن حجم السائل الذي سيخرج من الصمام لابد أن يستبدل مكانه بالهواء . ووجود الغطاء يمنع دخول الهواء وبالتالي سيضطر لان يدخل من الصمام وهذا يجعل إعادة خلط للطبقتين وبالتالي تكون كفاءة الفصل غير دقيقة.
3. What is the role of sodium carbonate in the extraction of caffeine from tea leaves? .......... (Salting out)

1. Decreases the solubility of organic compounds in the saturated aqueous phase.
2. Decrease the solubility of the organic and aqueous phases in each other, thus improving their separation.

4. What can you do if you do not know which layer is which in an extraction procedure? OR How could you test to determine which was the aqueous layer?

Drop a small amount of water into the neck of the separatory funnel. Watch it carefully: if it remains in the upper layer, that layer is the aqueous layer. If it sinks to the bottom of the upper layer to the interface between the two liquids, the bottom layer is the water layer.

Questions

1. Why was it important to open the stopcock every 10 seconds of shaking?
2. 200 mg of an organic solute is dissolved in 10 mL of water. How many mg of the organic solute will remain in the water after it has been extracted using 10 mL of diethyl ether? The KD for the system is 4.0 (diethyl ether solubility over water solubility.)

Thin layer Chromatography (TLC)

A. TLC is generally used as a qualitative analytical technique for:

1. determining the number of components in a mixture.
2. checking the purity of a compound.
3. following the course of a reaction.
How will the following affect the TLC separation?

A. Having too much solvent in the developing chamber so that it is surface extends above the origin (pencil line)?
   Or
   Why must the spotted marks remain above the level of the liquid in the beaker?
The solvent must not be above the pencil line used to guide the spotting of the samples. If it is, the samples will dissolve into the reservoir of solvent instead of traveling up the plate.

B. Which compound, ortho- or para-nitroaniline, should be more strongly adsorbed onto the silica gel? Why is this?
   para-nitroaniline (more polar) strongly adsorbed onto polar silica gel surface.

C. What will the result be of spotting too much compound onto the TLC plate?
   Larger spots result in tailing and overlapping of close spots.

D. Polarity of the solvent being too high
   The sample has migrated up the plate almost as fast as the solvent with little separation of the components.

F. Stationary phase is too active
   Polar compounds stick to the adsorbent more readily, and thus do not travel as far and have a lower value for Rf.

1) Consider the following silica gel TLC plate of compounds A, B, and C developed in hexanes
Determine the R\textsubscript{f} values of compounds A, B, and C run on a silica gel TLC plate using hexanes as the solvent

The distance between the origin and the solvent front: 2.75 cm. Then measure the distance between the origin and the center of spot A: 0.80 cm.

R\textsubscript{f} for compound A is 0.80/2.75 = 0.29.

R\textsubscript{f} for compound B is 2.20/2.75 = 0.80.

The R\textsubscript{f} for compound C is 1.70/2.75 = 0.62.

**Which compound, A, B, or C, is the most polar?**

Compound A is the most polar because it does not travel as far as the other two compounds. Remember, polar compounds stick to the adsorbent more readily, and thus do not travel as far and have a lower value for R\textsubscript{f}.

**How would the R\textsubscript{f} values change if eluted with hexanes using an alumina TLC plate?**

Alumina is more polar than silica. Therefore, each of the compounds would travel slower on an alumina TLC plate than on a silica TLC plate, the R\textsubscript{f} values for each of the compounds would be smaller.

**Consider a sample that is a mixture composed of biphenyl, benzoic acid, and benzyl alcohol. The sample is spotted on a TLC plate and developed in a hexanes/ethyl...**
acetate solvent mixture. Predict the relative R f values for the three compounds in the sample

Biphenyl is an unsaturated hydrocarbon, and therefore is the least polar and will have the largest R f value of the three compounds. The benzoic acid - an acid - would be the most polar and therefore will have the smallest Rf value. Benzyl alcohol is between these two compounds in polarity and will have an Rf value between the two.

Which dye would be more soluble in n-propyl alcohol, the blue or the yellow?

Blue dye more soluble than yellow dye in n-propyl alcohol and this was clear in paper chromatography experiment.

Why must you use a pencil not ink to mark the origin of the spots on the chromatography paper?

Synthesis of aspirin

1. Calculate the theoretical yield of aspirin, in grams, assuming that you start with 2.0 grams of salicylic acid and 5.0 mL of acetic anhydride. The density of acetic anhydride is 1.08 g/mL.

2. If 1.9 grams of aspirin were obtained in this experiment, what would be the percent yield?

3. What are the two simple techniques that can be used ensure the purity of the major formed product (aspirin)?

   a. Thin layer chromatography.
   b. Melting point.
   c. Ferric chloride solution.

4. Write the equation for the reaction which occurs when acetic anhydride is mixed with water.

5. What was the purpose of adding the sulfuric acid?
   Used as a catalyst (Used to speed up the reaction).
6. Why is the actual yield of a reaction almost always smaller than the theoretical yield? Can the percent yield of a chemical reaction over exceed 100%?

Actual yield < Theoretical yield. Why?

1. Not all the reactants may react.
2. Some of reaction are reversible (not all reactant → product).
3. There may be side reaction.

Actual yield > Theoretical yield: This occurs when the product is not dry (contains water).

7. Addition of cold water to the reaction flask during the synthesis of Aspirin

1. to hydrolysis excess acetic anhydride to acetic acid.
2. To complete the precipitation of aspirin (insoluble in water)

Synthesis of Aspirin

Here is a balanced equation for the synthesis of aspirin:

\[
\text{Salicylic acid} + \text{Acetic anhydride} \xrightarrow{H_3PO_4} \text{Aspirin} + \text{Acetic acid}
\]

Functional groups involved: Salicylic acid is both a carboxylic acid and a phenol. Acetic anhydride is an anhydride. Aspirin is a carboxylic acid and an ester. Acetic acid is a carboxylic acid.
The mechanism of this reaction is: (Just to know)

\[
\text{H}_3\text{PO}_4 + \text{CH}_3\text{C}O\text{C}O\text{C}CH_3 \rightarrow \text{H}_2\text{PO}_4 + \text{CH}_3\text{C}O\text{C}O\text{C}CH_3
\]
Boiling point and Distillation

1. Why must the thermometer be positioned exactly in the distilling head so the top of the mercury bulb is even with the bottom of the outlet to the condenser?

Positioned there in front of the condense the thermometer records most accurately the temperature of the vapor that is condensing and running down the condenser for collection.

2. What is the effect on the boiling point of a solution (e.g. water) produced by a soluble non-volatile substance (e.g. sodium chloride)? What is the effect an insoluble substance such as sand or charcoal?

A soluble, nonvolatile substance such as sodium chloride will raise the boiling point of an aqueous solution.

An insoluble substance will have no effect on boiling point of a solution.

3. What is the effect of each of the following on the observed boiling point?

a) The presence of a non-volatile impurity?

A soluble, nonvolatile substance will raise the boiling point of an aqueous solution.

Insoluble substance will have no effect on boiling point of a solution.

b) If you place the thermometer bulb too high (is not kept moist with condensate?)

The vapor will not reach to the thermometer bulb, Boiling point lower than should be.

4. Why it is important that cooling water enters at the lower end and exits at the upper end of the condenser jacket, and not vice versa?

*This ensures that the condenser jacket is always full of water.
To increase efficiency of cooling.

5. Where is the position of the thermometer bulb in the distillation apparatus?

The top of the thermometer bulb is even with the bottom surface of the outlet port of the connecting adapter.

Or

The bulb of the thermometer should be below the opening of the side arm of the distillation head to measure the most accurate temperature.

6. What effect does reduction of the external pressure have on the boiling point?

The boiling point will be lower because a lower vapor pressure will result in boiling.

7. Explain the relationship between the boiling point and atmospheric pressure

When the atmospheric pressure goes up so does the boiling point.
When the atmospheric pressure drops the boiling point goes down

8. What is the reason that liquids have different normal boiling points?

Intermolecular forces are different for different molecules. When the intermolecular forces are weak the boiling point are low. When the intermolecular forces are strong the boiling points are high

9. What is normal boiling point?

The normal boiling point is the temperature at which the vapor pressure equals one atmosphere.

10. During distillation why the distilling flask should be filled to two thirds of its capacity only?

If the flask is too full, liquid easily bumps over into the condenser and is not distilled.

11. How does the simple distillation work?

When the solution is heated, the molecules leave the liquid phase and enter the gas phase. The vapor reaches the thermometer and the temperature reading increases rapidly. The gaseous molecules reach the condenser and return to the liquid state and drain into the receiving flask. With each successive fraction, the temperature of the distillate vapor
rises. To isolate distillate fractions of different boiling point, the experimenter must change the receiving flask at selected intervals.

12. Mention the types of distillation

1. Simple distillation
2. Fractional distillation
3. Vacuum distillation
4. Steam distillation

13. Mention the Uses of simple distillation

1. Determination the boiling point of pure liquid.
2. Purification of liquids (separation of solid non-volatile impurities from a liquid).
3. Separation of any two miscible liquid that has boiling point difference > 80°C.

14. There is equilibrium during distillation

1. Rate of distillation 1 drops/sec.
2. Always there is a drop of condensate hang to the thermometer bulb.
Steam distillation

1. Explain why the clove oil could not be distilled directly from the cloves.

Clove oil contains component molecules whose boiling points are quite high. Heating them to their boiling point would be destructive.

2. Explain the principles by which steam distillation works.

Some organic compounds are immiscible with water. Usually these compounds have a low vapour pressure. After mixing them with water, however, the mixture will distil when the sum of the two vapour pressures reaches atmospheric pressure. It follows, then, that this must happen below the boiling point of water. This process is known as steam distillation.

3. Suggest a reason why the clove oil is much more soluble in non-polar solvents (than in water).

The component molecules found in clove oil are often relatively non-polar and will therefore be more soluble in the non-polar solvent than in water.
Quiz(1)

Student Name:……………………………………………………………………

Choose the most correct answer

1. If a compound boils at 100 degrees at 760 mm, its boiling point at 725 mm will be:
   a) lower                  b) higher                  c) the same                  d) cannot predict

2. Which of the following has the lowest vapor pressure?
   a) Water.                b) 0.1 M NaCl (aq).      c) 0.5 M NaCl(aq).          d) all have the same vapor pressure.

3. How will an increase in temperature affect vapor pressure?
   a) Increase              b) decrease              c) stay the same

4. The purpose of putting boiling chips into a distillation flask is to …
   a) Reduce the vapor pressure.              b) Increase the number of theoretical plates.
   c) Increase the vapor pressure            d) prevents superheating.

5. In a distillation of a mixture of two components, the first compound to condense into the receiving flask will be…
   a) the one with the lower melting point.      b) The one with the higher melting point.
   c) The one with the lower boiling point.      d) The one with the higher boiling point.

6. In general, what is the purpose of simple distillation?
   a) To purify solid materials.                b) To separate two immiscible liquid.
   b) To separate liquids from one another.    c) To purify dyes

7. An important part of the distillation apparatus that condenses vapor to a liquid is the…
   a) Boiling round bottomed flask             b) receiver              c) condenser              d) Adapter

8. The temperature at which the vapor pressure of a liquid exactly equals the atmospheric pressure is…
   a) Boiling point                        b) melting point            c) partial pressure       d) distribution coefficient
9. Which physical property would be affected most by changes in the atmospheric pressure?
   a) Melting point  b) boiling point  c) refractive index  d) molecular weight

10. The type of intermolecular force in CH₃OCH₃…
   a) ion–ion  b) dipole–dipole  c) hydrogen bonding  d) dispersion forces

11. In the boiling point, if the stream of air bubbles is rapidly escaping from the open end of the capillary tube. Then the vapor pressure of the liquid is:
   a) Equal the atmospheric pressure.
   b) Higher than the atmospheric pressure.
   c) Lower than the atmospheric pressure.

12. Boiling point:
   a) Directly proportional to the atmospheric pressure
   b) Inversely proportional to the atmospheric pressure
   c) Directly proportional to the intermolecular forces
   d) {a & b}  e) {a & c}

13. The substance does not loss its identity after carrying out:
   a) the physical prosperities  b) the chemical prosperities
   c) {a & b}  c) none of all
Answer the following Questions:

1. **What does the atmospheric pressure affect the melting point?**
   No effect

2. **What is the purpose of determining the melting point in this lab?**
   A) To determine the identity of an organic solid.
   B) To determine the purity of an organic solid.

3. **Why is this method not used for finding the melting point of inorganic ionic compounds?**
   Ionic compound … Lattice strong … melting point will be high.

4. **What is the purpose of recrystallization?**
   It is the primary method of purifying crude solid compounds

5. **How do you get rid of soluble and insoluble impurities?**
   Insoluble impurities: They are removed through the method of hot filtration
   Soluble impurities: Suction or Vacuum Filtration

6. **What is the role of sodium carbonate in the extraction of caffeine from tea leaves?**
   1) Sodium Carbonate is used to remove the acidic tannins by converting them to water soluble salts.
   2) Decreases the solubility of organic compounds in the saturated aqueous phase.
   3) Decrease the solubility of the organic and aqueous phases in each other, thus improving their separation.
Quiz(3)

Student Name: ………………………………………………………………………………………………

Answer the following Questions:

1. Why is it recommended to use no more than the minimum amount of solvent than is necessary to dissolve the solid being recrystallized?

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2. Why fluted filter paper used for a hot filtration?

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3. How do you separate the purified solid crystals from the surrounding liquid?

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4. What is the role of sodium sulfate in the extraction of caffeine from tea leaves?

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5. Why was it important to open the stopcock every 10 seconds of shaking?

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Good luck