CHEM 2219: Exp. # 4 Recrystallization of Acetanilide

Objective: In this experiment you will learn to remove impurities from a compound by recrystallization.* First the best recrystallization solvent will be determined for the compound, then hot gravity filtration and vacuum filtration will be used to separate the compound from the impurities. The melting point range for the original and purified crystals will be determined in order to verify the procedure was successful.

* Recrystallization is a common technique used to purify solids. A compound is considered impure if there are minor components of another compound mixed in with it; or, when there is a mixture of two or more components and only one of the components is desired. The undesirable components are considered impurities. The melting point (MP) is a physical property of a solid also used for the purpose of determining purity.

Reading Assignment: MTOL, pp. 110-118 (recrystallization); and,
OCLT, pp. 155-199 (crystallization) p372 (testing solvents for crystallization), and
p. 374 (single solvent crystallization).
Also on **CANVAS** read and answer prelab questions for **– Recrystallization**

Concepts:
Hot Filtration, Impurities, Oiling Out, Recrystallization, Solubility, Vacuum Filtration

Chemicals:
acetanilide, acetone, activated carbon, dichloromethane, ethanol, hexane and water

Safety Precautions:
Wear chemical splash-proof goggles and appropriate attire at all times.
Acetone, dichloromethane, ethanol and hexane are flammable liquids.
Boiling water and steam can cause severe burns. Hot glassware looks like cold glassware.
Always use the proper tongs when handling hot glassware in order to avoid spills.

Materials:
balance, beakers (100 ml & 250 ml), beaker tongs, copper pot, crucible tongs, disposable pipet and bulb, erlenmeyer flask (50 ml & 125 ml), filter flask, filter paper (3 cm & 9cm), glass stirring rod, 3cm Hirsch funnel, hotplate, melting point apparatus, microspatula, mortar & pestle, stemless funnel, 5 test tubes, test tube clamp, test tube rack, vacuum tubing, watchglass, and weighing paper

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At your station, you should have the following:

1.) Test tube rack with 5 test tubes labeled 1-5 containing known solvents.
2.) A vial containing pure acetanilide.
3.) 1 mini-test tube for measuring ground up pure acetanilide. (optional)
4.) A hot plate (200 ^{o}C) with a hot water bath – a 250 ml beaker with ~150 ml of water
5.) Beaker tongs and crucible tongs
6.) A small weigh boat

Procedure for Determination of the Correct Recrystallization Solvent:

1. Observe and record the following:
   a. Color and appearance of the pure acetanilide.
   b. Color and appearance of the solvents.
      (e.g., clear colorless liquid, cloudy orange liquid, etc.)

2. Using the mortar and pestle from your drawer, grind up the pure acetanilide. Transfer the powder to the small weigh boat.

3. Using the microspatula spoon, add acetanilide to the mini-test tube to the fill line.

4. Add the acetanilide from the mini-test tube to the first solvent.

5. Repeat steps 3 and 4 for the remaining solvents.

6. Place a rubber stopper in the end of the solvent test tube and shake to dissolve the solid.

> ***Note:*** If the sample does not readily dissolve, you may also try stirring the solution with a glass stirring rod. If it still does not dissolve, the sample is not soluble in that particular solvent.

7. Record in a table whether or not the acetanilide dissolves at room temperature. (A sample table has been provided on page 6.)

> ***Note:*** The sample will dissolve in two of the solvents, not dissolve in two of the solvents, and depending on the ambient temperature may partially dissolve in one of the solvents. If the sample dissolves at all at room temperature, then the solvent is not suitable for recrystallization and does not need not be tested in the hot water bath.

8. Using the test tube clamps from your drawer, place the test tubes containing the remaining two potential recrystallization solvents in the hot water bath.

> **(*Caution*: Some of the solvents have low BP and will evaporate rapidly in the hot water bath. Do *not* leave a test tube in the hot water bath once the solvent starts boiling and evaporating off as hot acetanilide might spit out of the test tube and burn you.)**

9. Record in a table whether or not the acetanilide dissolves in the solvent at its boiling point.

> ***Note 1:*** In the Soluble at BP column, record "X"s for the solvents that were not tested
> ***Note 2:*** The acetanilide will dissolve in only of the samples at its BP.
> This is the solvent you want to use for recrystallization.

10. Remove test tubes from the hot water bath and place them in the test tube rack to cool.

11. Record the identity of the solvent you have chosen as your recrystallization solvent.

Note: The solvent in which the acetanilide did not dissolve at RT, but was soluble at its BP is the recrystallization solvent you want to use. Record which solvent was determined to be the best. If you are unsure, please check with your TA or instructor.

12. Verify your choice of recrystallization solvent with a TA or an instructor.

13. Clean up this portion later after the unknown in solution has been completely added to the hot filtration system. Dispose of all chemicals appropriately. (*You may proceed to the next portion while the test tubes are cooling and come back to this step*.)

Recrystallization of Acetanilide Procedure

At your station, you should have the following:

1.) A hot plate (200 °C) with a hot water bath:
A 250 ml beaker with ~150 ml of water
2.) A vial with unknown number containing acetanilide with a contaminant.
3.) A hot filtration system:
A 250 ml beaker
A stemless funnel.
A 9 cm piece of filter paper fluted.
A 4 inch watch glass.
4.) A disposable pipet and bulb
5.) A copper pot for making an ice bath

In your drawer:

1.) 125 ml Erlenmeyer flask
2.) 50 ml Erlenmeyer flask

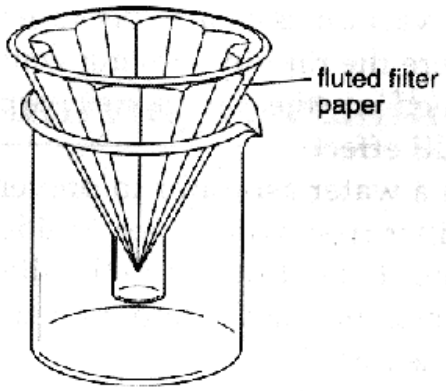

Figure 1: Hot filtration system.

Procedure for Recrystallization of Acetanilide:

1. Verify that the hot filtration system is set up as shown in Figure 1.

2. Add ~20 ml of hot recrystallization solvent to the 250 ml beaker, pouring it over the fluted filter in the stemless funnel. Place the 250 ml beaker on the hot plate. Place the 4" watch glass on top of the funnel in the beaker.

3. Add ~30 ml of hot recrystallization solvent to a 125 ml Erlenmeyer flask and place it on the hotplate.

4. Record your unknown number. Record physical properties (color / consistency) of the unknown.

5. Add all of the unknown to a clean mortar. Using a pestle, grind up the unknown.

6. Return a small amount of the ground unknown to its disposable vial to be used later for a melting point determination.

7. Transfer the remainder of the ground unknown to a tared weighing boat. Weigh it. Record the mass to the nearest mg (0.001 g). [***Note:*** *The mass should be between 1.3 – 1.8 g. If not contact a TA*.]

8. Pour the solid into a 50 ml Erlenmeyer flask.

9. Determine the amount of solvent you will need.* Show calculation and record volume determined.

***Solvent determination:** You need 20 ml of solvent for every gram of acetanilide. (Based on the ratio 1g/20ml for the solubility of acetanilide in the boiling solvent.)

10. Using crucible tongs to pick up the hot 125 Erlenmeyer flasks containing the boiling solvent, add the determined amount of hot solvent from the 125 Erlenmeyer to the 50 ml Erlenmeyer. Swirl the solution in the 50 ml Erlenmeyer to help dissolve the solid. Place both flasks on the hotplate.

Note: Some material will remain undissolved. This is the insoluble impurity intentionally added to the acetanilide unknowns.

11. When the solution containing the sample is boiling, remove the watch glass from the hot filtration setup. Use crucible tongs to pick up the 50 ml Erlenmeyer flask. Pour the sample from the 50ml Erlenmeyer rapidly over the filter, being careful not to overflow the system and not to pour the solution between the filter and the funnel. Replace the watch glass cover.

Note: If the sample is poured too slowly, it will crystallize out along the neck of the Erlenmeyer flask and / or on the filter paper. If this happens the sample may need to be crushed with a microspatula in order to get it fine enough to dissolve when additional hot solvent is added.

12. Place the 50 ml Erlenmeyer flask on the lab bench. Pour ~10 ml of hot solvent from the 125 ml Erlenmeyer flask into the 50 ml flask. Swirl the solvent around in the 50 ml flask to dissolve any residual sample. Place both flasks on the hotplate. When the solvent of the sample solution has passed through the filter paper, use the additional hot solvent in the 50 ml Erlenmeyer flask to rinse the filter paper as before. Place the empty 50 ml Erlenmeyer flask on the bench top. Place the 125 ml flask back on the hotplate.

13. Wait 5 minutes. Check the filter paper for any residual acetanilide.
    If any shiny crystals are visible, using a disposable pipet, rinse the crystals with more hot solvent and wait 5 more minutes. Repeat until no crystals are visible.
    If not, remove the watch glass and the funnel and set them on the lab bench.
    (Be careful, they are hot!)
    The Erlenmeyer flasks can also be removed from the hotplate and set aside.

14. Move the 250 ml beaker to the center of the hotplate. Turn the hotplate up to 300 ^{o}C. Allow the solvent in the beaker to evaporate down to the volume originally determined to be needed to dissolve your sample.

***Note:** This would be an excellent time to clean up the glassware and equipment used in Part I. Pour the liquids out of the test tubes into the waste container. Rinse each test tube with acetone until there is no solid residue remaining. Place test tubes in rack. Place the rack back in your desk. Dispose of the vials for "the unknown" in the glass waste.

15. When the volume has been reduced adequately, using beaker tongs, remove the 250 ml beaker from the hotplate and set it on the desktop to cool. Cover the beaker with the watch glass. Crystals should form upon cooling.

Note: If no crystals form on cooling, you may still have too much solvent present. Try stirring and scratching the beaker with a glass stirring rod to induce crystallization. If this does not work, place the beaker back on the hotplate and reduce the volume of the solvent. Then repeat step 15.

16. Make an ice bath using the copper pot on the bench top. Add ice. (*The ice is in a styrofoam ice chest located near the safety shower*.) Add a small amount of distilled water. (*The ice bath should be the consistency of a thick slushie but not too full so your containers don't float and tip over*.)

17. When the solution is at ~RT, place the beaker in the ice bath to further reduce the compound's solubility. Also place a 100 ml beaker with ~50 ml of solvent in the ice bath to cool for use in rinsing the crystals during the final vacuum filtration step.

18. A vacuum filtration apparatus should be setup at your station (as shown in Fig. 2) using the 250 ml filter flask, the 3.0 cm Hirsch funnel and the 3.0 cm filter paper. Make sure the seal is tight. Place the filter paper on the funnel. Seal the paper with a small amount of cold solvent. Make sure all of the holes in the funnel are covered by the filter paper and the edges are not curling up. Before connecting the hose to the flask, test the vacuum by attaching the hose to the vacuum and placing your thumb over the hole on the other end of the tube and turning the vacuum up until you can feel it. Once the vacuum is working, attach the hose to the flask. Seal the filter paper again using the cold distilled water.

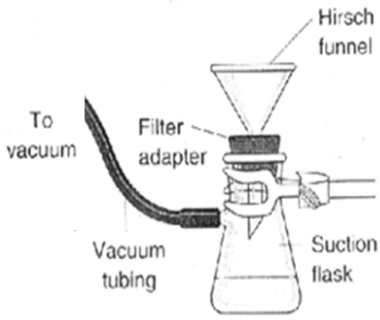

Figure 2: Vacuum filtration system.

19. Pour the cold solution containing the white crystals over the filter. Be careful not to overflow the funnel. To wash the crystals with cold solvent, disconnect the vacuum hose, add cold solvent, and then reconnect the vacuum.

20. After the final rinsing, allow the vacuum to run for 5-10 min* to dry the crystals. Periodically run the microspatula through the crystals to "unclump" them. Be careful not to tear or lift the filter paper.

> ***Note:** You can run the MP of your initial unknown while the recrystallized unknown is drying.*

21. Once the crystals are dry, disconnect the vacuum hose from the flask before turning off the vacuum.

22. Be careful when removing the funnel not to lose any crystals. Take the funnel and a microspatula to the balance. Transfer the crystals from the filter paper to a tared weighing boat. Weigh the crystals. Record the mass to the nearest mg (0.001 g).

23. Record the model name and number of the MP apparatus used. Consult handout for MP determination instructions. Determine and record the MP range for both acetanilide unknown samples: prior to and after recrystallization.

24. Dispose of chemicals appropriately.* Clean and return all glassware and equipment to their original locations. (That is, leave on the benchtop at your station, two 250 ml beakers, the stemless funnel, the 4" watchglass, the pipet bulb, the 250 ml filter flask and the 3 cm Hirsch funnel.
The mortar and pestle and Erlenmeyer flasks go back in your drawer.)

> ***Note:** If you would like to keep your recrystallized product, consult your instructor.*

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Observations:

Color & Appearance of Pure Acetanilide: _____

Solvent	Color / State	Soluble at Room Temperature (Yes / No)	Soluble at BP of Solvent (Yes / No / X)
1. Acetone			
2. Dichloromethane			
3. Ethanol			
4. Hexane			
5. Water			

Solvent Chosen for Recrystallization of Acetanilide: _____

Post Lab:

1. Calculate a **Simple %Recovery** for the recrystallization of acetanilide.
   Simple %Recovery = (Final Mass / Initial Mass) x 100
2. Calculate a **%Recovery based on Solubility** of the acetanilide in the solvent.
   First determine the maximum percent recoverable:

   $$\textbf{Max \% Recoverable} = \frac{[((y \text{ ml})(1 \text{ g} / 20 \text{ ml})) - ((y \text{ ml})(1 \text{ g}/100 \text{ ml})]}{x \text{ g}} \times 100$$

   where x grams = initial number of grams
   y ml = number of ml determined

   Then determine the maximum mass recoverable:
   Max mass recoverable (g) = Max % Recoverable x Initial Mass

   Then substitute the maximum mass recoverable for the initial mass in the % Recovery eqn:

   % Recovery$_{\textbf{(solubility)}}$ = (Final Mass / Max Mass Recoverable) x 100
3. Calculate % Errors for both MPs.
4. Tabulate the results. Show sample calculations.
5. In your conclusion, discuss
   a. How recrystallization purified your sample.
      (For example, note any color changes and changes in the melting point ranges and melting point averages.)
   b. Whether the experiment was successfully performed and / or any problems that arose during the experiment and your recommendation for how to avoid those problems if you were to redo the experiment.