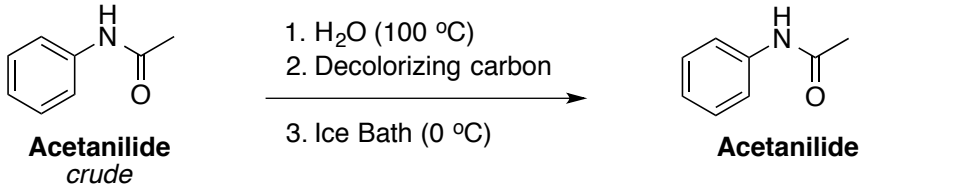
Experiment 1: Recrystallization of Acetanilide

Reading Assignment
Mohrig 2 – 4 (Glassware, Reagents, & Heating) & 14 – 15 (Melting Point & Recrystallization)

The purification of organic compounds is a tedious, yet vital, part of synthetic organic chemistry. Successful organic synthesis requires very pure starting materials to avoid complications from impurities. When possible and practical, solids are purified *via* recrystallization or sublimation and liquids *via* distillation. In this experiment, students will purify crude acetanilide *via* recrystallization from water (**Scheme 1**).



Acetanilide
crude

Acetanilide

crystalline, ____% recovery

Scheme 1. Recrystallization of acetanilide from water.[1]

The purity of the crude and recrystallized acetanilide will be assessed by **melting point**. Recall that colligative properties predict that impurities lower melting points and increase boiling points. Purity may also be apparent in the appearance of the solid before and after the experiment. The product has fewer impurities and a more ordered structure.

The crude solid is dissolved in the smallest possible amount of solvent of choice; in this case the solvent is water. Acetanilide has a much higher solubility in hot water than in cold water. The purified solid will not recrystallize later in the experiment if too much hot solvent is added in the beginning. Activated charcoal is then added to remove **colored impurities**. These impurities are often polar organic compounds that have an affinity for activated charcoal. Any **insoluble impurities** (including those that have adsorbed onto the charcoal) are removed during the **hot filtration** step, while acetanilide remains in solution. This solution is gradually cooled in an ice bath to induce precipitation. Any **soluble impurities** remain in solution during the **cold filtration**, while the purified solid remains on the filter paper.

Thus, the general steps of recrystallization are as follows:

- Choose a good recrystallization solvent
- Dissolve the sample in the *minimum* amount of boiling solvent
- Hot filtration to remove insoluble impurities
- Cool the solution to induce crystallization
- Cold filtration to separate the solid from the solution (mother liquor)
- Wash the solid with a small amount of cold solvent
- Dry the solid to remove traces of solvent

Finding the proper **recrystallization solvent** has to be determined experimentally and can be tricky if there is no literature precedent for the compound (luckily there is for acetanilide!). A successful recrystallization requires that the compound be soluble at the solvent's boiling point and insoluble at low temperature. The masses of recrystallized product, m_{recrys}, and the original crude starting material, m_{crude}, are used to calculate the **percent recovery of recrystallization** according to **eq 1**.

$$\% \text{ Recovery} = \frac{m_{recrys}}{m_{crude}} \times 100 \qquad \textbf{(1)}$$

[1] This scheme should appear in the "purpose" section of the lab notebook.

A **theoretical recovery** can be calculated if the solubilities at a cold temperature, S^c, and solubility at high temperature, S^H, are known. Since the predicted mass recovery would be the difference between the hot and cold solubility, the relationship can be expressed in **eq 2**. A theoretical recovery can be calculated from an actual experiment by combining **eq 1** and **eq 2**, using m_{crude} in place of S^H in the denominator of **eq 2**.

$$\text{Theoretical Recovery} = \frac{S^H - S^c}{S^H} \times 100 \qquad (2)$$

Note that the percent *recovery* and theoretical *recovery* are different from the theoretical *yield*. Theoretical *yield* applies to a chemical reaction and *recovery* refers to a physical process.

Notebook Preparation – See sample notebook page on eCommons.
Preparing a good lab notebook goes beyond just writing things down! Make sure you understand what you're writing and have a ***game plan*** *for what to do when you get to lab. Attending lecture and office hours are key. You cannot use the handout in lab.*

- *Purpose*: one-sentence description plus **Scheme 1**.
- *Reagent Table*: amount (mg or mL), mmol, MW, bp or mp, density, and one-word hazards (see safety table) for each of the chemicals used. Leave space in the table to write the actual amounts of starting materials used.
- *Procedure*: Hand-written, step-wise procedure in your own words (short phrases, not complete sentences, using bullet points or numbers). Pictures encouraged. Keep this section as brief as possible, ideally under a page or two.
- *Safety & Clean-up* – Copy the table at the end of the procedure into your notebook.

PROCEDURE – *Students work in pairs.*

Dissolving the Sample. Place *approximately* 2 g of crude acetanilide in a 125-mL Erlenmeyer flask. Record the actual mass weighed. Add 35 mL of water plus two boiling chips and bring the suspension to a boil on a hot plate (hot plates should *never* go past medium setting). Stir the system frequently with a glass rod. Allow the solution to boil for a few minutes then, if necessary, add more water (up to 5 mL max) drop-wise until all solid dissolves. Instead of dissolving, some material may "oil out" and appear as oily droplets. In this case, treat it as dissolved solid and proceed. Adding more water may decrease the percent recovery. *Record the total amount of water added in the reagent table.* When all the acetanilide dissolves, remove the flask from the hot plate. If crystals deposit from solution as it cools, add 1-3 mL water to re-dissolve. Slowly add a dash of activated charcoal (do not add charcoal to a boiling solution!).** Place the flask on the hot plate again and bring the system to a second boil. Stir while heating to prevent bumping (violent boiling & splashing) and set up the hot filtration apparatus.

Hot Filtration Place one or two boiling chips and approximately 10 mL of water into another 125-mL Erlenmeyer flask. Cut or find a small piece of copper wire, about an inch in length, bend it into a U and place it over the lip of the flask. This will provide space for steam to escape between the flask and funnel. Place a short-stem funnel equipped with a fluted piece of filter paper on top of the flask, and place the complete apparatus onto the hot plate.

When the funnel is heated by the steam, discard the remaining water (but keep the boiling chips) and quickly begin the hot filtration by pouring the acetanilide-charcoal suspension into the fluted filter paper with the aid of the glass rod to direct the solution into the funnel. Fill the funnel no more than half full at any given time. Maintain the temperature of both solutions to prevent premature crystallization in the funnel, periodically placing the flasks back on the hot plate. If substantial crystallization of acetanilide occurs in the filter, you may rinse the crystals down with small portions of boiling water. Alternatively, you may stop the filtration and transfer as much acetanilide as possible from the filter to the Erlenmeyer flask and restart from **.

Cooling Down After completing the hot filtration, discard the filter paper in solid waste and allow the flask containing the mother liquor to cool to *room temperature* then place it in an ice-water bath. If the system cools down too quickly, small crystals form and adsorb a large amount of impurities from the mother liquor. Also place 5 mL of distilled water in the ice bath. This will be used later to wash the crystals. Allow crystals to form for 10 minutes (note initial time of crystal formation as it may not happen immediately). Do not disturb the flask once crystal formation has begun. If no crystals have formed after five minutes on ice, scratch the bottom of the flask with a glass stir rod.

Cold Filtration After crystallization is complete, collect the crystals by vacuum filtration using a 125-mL filter flask, a Buchner funnel, and a *pre-weighed* piece of filter paper. The filter paper should cover all the holes on the Buchner funnel without folding up to the walls. Position the filter paper on the funnel, then run the vacuum on and wet the filter paper with 5-10 mL of *cold* water. This will adhere the paper to the funnel and prevent it from moving during the cold filtration. Filter your cold acetanilide-water mixture by pouring it into the Buchner funnel with the aid of a glass rod. To help the transfer of the solid acetanilide, gently swirl the contents of the Erlenmeyer flask then pour the suspension into the funnel. Dispose of the mother liquor at the end of lab.

Washing and Drying the Solid Once you finish filtering the whole solution, and the liquid stops dripping from the funnel, turn off the vacuum and add a few milliliters of ice-cold water to the funnel to wash the crystals. Stir the solid very gently with a glass rod or a spatula without touching the filter paper. Turn the vacuum on and press the crystals with a spatula (tip should be slightly bent) to remove as much water as possible. Let the solid dry on the filter with the vacuum on for 5-10 minutes, then take a small sample for melting point analysis. Dry the solid on a porous plate; take the melting point of the crude solid simultaneously. Keep the vacuum on for an additional 30 minutes, until solid is dry. Transfer the solid and filter paper to a pre-tared watch glass.

Weigh the dried solid and calculate the mass of pure acetanilide by difference. Calculate percent recovery and *record observations*. If the recovery is greater than 100%, the solid requires more drying. Put the filter paper back in the funnel and turn the vacuum on.

Clean-up and Waste Procedures	**Safety Hazards**
* *Solid waste*: Filter papers, used capillaries, and product * *Liquid waste*: Mother liquor * Wipe down all bench tops, wash glassware, and return equipment to proper place in an organized fashion.	* Be careful with hot glassware. Use paper towels or cloth to handle. Do not use clamps. * Do not let acetanilide come in contact with eyes, mouth, or skin (irritant).

Introduction: Pre-lab Questions
Turn in typed responses in the beginning of lab. Your TA will keep this until the report due date. Use complete sentences and keep your responses as brief as possible.

1. List the basic steps in the recrystallization of acetanilide. Include the identity of the solvent that will be used and what is added before hot filtration (see the "general steps" and/or the paragraph headings in the procedure).

2. What functional groups are present in acetanilide? Are these groups considered polar or non-polar? Use this information to explain why water is a good recrystallization solvent for acetanilide.

3. After hot filtration removes insoluble impurities, the remaining solution is cooled in an ice bath without being disturbed. Explain what happens during the crystallization process and what happens if crystals form too quickly.

4. Why should a minimum amount of hot solvent be used for recrystallization? Why must the recrystallized solid be washed with *cold* solvent?

5. What effects do most impurities have on the melting point of organic compounds (recall what you have learned about colligative properties)?

6. The solubility of acetanilide in hot water is 5.5 g/100 mL at 100 °C and its solubility in cold water is 0.53 g/100 mL at 0 °C. What is theoretical percent recovery from this recrystallization experiment, assuming you will use exactly 2.00 g of crude acetanilide and 35.0 mL of water (*hint*: see equations 1 and 2)? Show your work (typed or in pen).

Results: In-lab Questions
Copy an abbreviated version of these questions into your notebook to prevent you from missing data. Complete before leaving lab then type responses in complete sentences for the report.

1. Report the **actual mass of crude acetanilide, amount of water, theoretical percent recovery, and mass of recrystallized acetanilide** in one or two complete sentences. Include **uncertainty (ILE's)** in each of your measurements (ex. 2.010 ± 0.0005 g). Re-calculate the theoretical percent recovery from *your* recrystallization experiment, taking into account the actual mass of crude acetanilide and the total amount of water used in your experiment (similar to pre-lab #6). You do not need to show your work.

2. Report the **percent recovery** of the recrystallization of acetanilide from water. Show your work (typed or in pen).

3. Briefly provide **experimental observations** with special attention to sources of potential product loss.

4. Report the **melting point ranges of the crude and recrystallized acetanilide**. Compare this to the literature melting point of acetanilide (higher or lower?) and *briefly* explain your results in terms of colligative properties.

Experiment Adapted from…Palleros, D. R. "Recrystallization of Acetanilide," *Experimental Organic Chemistry*, Wiley, New York, 2000, p. 80-85.

Exp 1 Recrystallization of Acetanilide Name _____
Due Date in Syllabus

Section Day _____ Time _____ TA Name _____

CHEM 108L GRADING RUBRIK - Use as cover page for report

SECTION	INSTRUCTOR COMMENTS	POINTS ASSIGNED
IN-LAB QUIZ		**/ 5**
LAB REPORT		
ABSTRACT One paragraph, four sentences: Purpose, procedure, main result(s), and conclusion(s).	NONE	0 / 0
INTRODUCTION Each pre-lab question is addressed in its own paragraph using complete sentences. Structures and calculations are hand-written, where appropriate. Obtain from TA on due date.		/ **30**
RESULTS The main results are stated, as outlined in the post-lab questions, using complete sentences.		/ **20**
DISCUSSION and CONCLUSION Reflection of whether results were as expected and whether it is supported by the theory behind the experiment. Three sources of error are stated and discussed in terms of the effect on the main result(s).	NONE	0 / 0
EXPERIMENTAL SECTION The experimental details (including final amount used and obtained) are *briefly* described in a few sentences.	NONE	0 / 0
NOTEBOOK PAGES Proper format: reaction scheme, chemical info table, procedure, waste and clean-up procedure.		/ **30**
NEATNESS AND ORGANIZATION Proper grammar, order, and format (see syllabus and Technical Writing Guidelines on eCommons for more detail)		/ **15**
	LAB REPORT TOTAL	**/ 100**