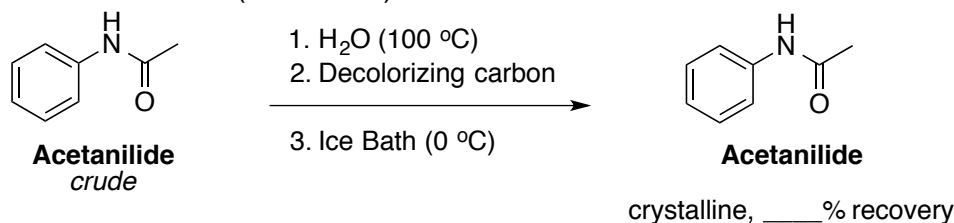


Experiment 1: Recrystallization of Acetanilide

Reading Assignment: Mohrig Sections 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 from water (**Scheme 1**).



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/freezing points and increase boiling points. Purity will also be apparent in the appearance of the solid before and after the experiment. The product has fewer impurities and a more ordered structure. Crude acetanilide looks like grains of brown rice, while pure acetanilide forms shiny crystals in cold water.

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 equation 1.

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

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

A **theoretical recovery** can be calculated if the solubility 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 equation 2. A theoretical recovery can be calculated from an actual experiment by combining equations 1 and 2, using m_{crude} in place of S^h in the denominator or eq. 2.

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

Note that the percent *recovery* and theoretical *recovery* are different from the theoretical *yield*. A theoretical *yield* would apply if a chemical reaction was taking place...and it's not!

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. You cannot use the handout or textbook 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 for each of the chemicals in the reaction, where applicable. 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 optional but 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 grams of crude acetanilide in a 125-mL Erlenmeyer flask. Record the actual mass weighed. Add 30 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 drop-wise until all of the solid (which may have just turned into oily droplets of melted acetanilide) dissolves. A *maximum* of 10 mL of additional water may be required to completely dissolve the acetanilide. Keep in mind that adding more water will 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 easily deposit from solution as it cools, add about 3 mL of water. 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. Due to the large surface area of charcoal, the suspension has a tendency to bump; avoid this by stirring while heating.

Hot Filtration After adding charcoal and before bringing the suspension to a second boil, set up the following apparatus. Place one or two boiling chips and approximately 10 mL of water into a 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. 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 has been heated by the steam, quickly discard the remaining water (but keep the boiling chips) and 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 and maintain the temperature of both solutions to prevent premature crystallization in the funnel. 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 hot 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 will not happen immediately). Do not disturb the flask once crystal formation has begun. If no crystals have formed after a few 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. Compare the yield with the amount of starting material and *record observations*. If the recovery is greater than 100%, the solid requires more drying. Consult the instructor for assistance. You will lose points if your recovery is greater than 100%!

Clean-up and Waste Procedures	Safety Hazards
<ul style="list-style-type: none"> * <i>Solid waste</i>: Filter papers, used capillaries, and product * <i>Liquid waste</i>: Mother liquor * Wipe down all bench tops, wash glassware, and return equipment to proper place in an organized fashion. 	<ul style="list-style-type: none"> * 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 tell you when to pick up your pre-labs. These original responses will be turned in with the lab report. You cannot change your answers. Use complete sentences and keep your responses as brief as possible.

1. Explain the purpose of recrystallization. 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" on the previous page and/or the paragraph headings in the procedure!).
2. Why should minimum amounts of solvent be used in any recrystallization?
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 why you should keep the flask still.
4. 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 g of crude acetanilide and 30 mL of water (*hint*: see equations 1 and 2)? Show your work (typed or in pen).

Results: Post-lab Questions – 5 points each

It is recommended to copy the questions into your notebook before lab. Complete in the lab notebook 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. Recalculate 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. Report the melting point ranges of the crude and recrystallized acetanilide. Compare this to literature values and *briefly* explain your results in terms of colligative properties.

Friendly reminder. Review the syllabus and lecture notes before coming to lab to be sure you have the requirements to perform the experiment. Thank you!

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 – Staple the report together in this order before coming to lab:		
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.		/ 35
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 <i>briefly</i> 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)		/ 10
LAB REPORT TOTAL		/ 100