LAB REPORT FOR EXPERIMENT #1: RECRYSTALLIZING A SOLID

Your name TA's name
Your Partner's name Lab Section

[Again, I'll be communicating in the brackets. I'll be making up the data as well!]

OBSERVATIONS:

A. SELECTING A RECRYSTALLIZATION SOLVENT

	Solubility Test (cold)	Solubility Test (hot)
Water	insoluble	somewhat soluble
pet ether	soluble	readily soluble
ethanol	fairly soluble	readily soluble
acetone	very soluble	readily soluble

After looking at the solubility of impure acetanilide in the four solvents, water seems to be the best choice as a recrystallizing solvent.

B. DECOLORIZING A SOLUTION OF IMPURE ACETANILIDE AND HOT FILTRATION

2. 1.5637 grams of impure acetanilide
5. 38 ml total solvent volume
6. Added tip of carbon black to mixture and mixture almost boiled over!
7. 28 ml total solvent volume

C. ISOLATING THE RECRYSTALLIZED PRODUCT WITH VACUUM FILTRATION

2. Chilled crystals in ice bath for 19 minutes.

Mass of watch glass and acetanilide	= 22.5478
Mass of watch glass	= 21.6903
Mass of recovered acetanilide	= 0.8575 g
Mass of impure acetanilide	= 1.5637 g
Percent recovered	= 54.8 %

D. DETERMINING THE MELTING RANGE

Melting range of recrystallized acetanilide	= 112 – 114 C
Melting range of impure acetanilide	= 105 – 111 C

1. Compare the melting ranges for the pure and impure acetanilide samples. What can you tell from these?

The impure acetanilide had a low, broad melting range while the recrystallized material had a sharper melting point much closer to the published values.

2. Compare the melting point of your purified acetanilide with that listed in the literature. Discuss any discrepancies. Are they significant?

The melting point obtained from the recrystallized product was slightly lower than the published value, probably due to water still trapped in the filter cake. More time drying would have helped give values that would be closer to the literature value.

3. Determine the percent recovery of pure acetanilide. Discuss possible losses of material during the recrystallization operation. Are there places you know that you lost a significant amount of material? What changes would you recommend to minimize such losses?

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The filtration step was the step in which most of the loss occurred. Crystallized starting material could be seen in the filter paper; this material had been dissolved earlier in the solution but when the hot liquid started cooling off, crystals came out of solution in the funnel. If the funnel and filter paper were kept wetter and hotter, this might not have happened.

CONCLUSION:

In this experiment a technique was used that will be used frequently throughout the semester: hot recrystallization. This technique allows an impure material to be purified and the risk of getting a lower yield, which isn’t necessarily a poor trade off. The purity of the material obtained can be correlated to the sharpness and veracity of the melting point data, which was seen.