Recrystallization and Melting Points

Recrystallization is an important method for the purification of solids. In this experiment you will be given an impure sample of an unknown organic solid which you will purify by recrystallization and identify by determining its melting point.

Required Reading:

Padías:

Laboratory Notebook (pp. 5 – 13), Basic Lab Techniques (pp. 16 – 30), % Recovery (p. 15) Solvents, Solubility (pp. 37 – 43)

Melting Point (pp. 47 – 53) Recrystallization (pp. 119 – 127)

Safety:
Be certain to frequently stir, or add a boiling chip to any liquid before heating it to prevent "bumping".

Procedure:
To a 125 mL Erlenmeyer flask add about 1.0 gram of a solid unknown. Add 10 mL of water and a boiling chip and heat the flask until the contents boil. Stir the hot mixture for about one minute. Continue to add water to the flask, 2-3 mL at a time and boiling for one minute after each addition until the unknown dissolves. Some insoluble impurities may remain and can be distinguished from the unknown by their appearance.

Filter the hot solution by gravity through a powder funnel containing fluted or folded filter paper into a third 125 mL Erlenmeyer flask. *The solution must remain hot during this operation.* It is always a good idea to moisten the filter paper with some hot solvent (water in this experiment) prior to filtration to prevent cooling. Rinse the filter paper with 2-3 mL of boiling water to dissolve any crystals that may have formed from cooling.

Allow the filtrate to cool slowly to room temperature. If no crystals have formed after cooling, scratch the inner wall of the flask with a glass rod to induce crystallization. Once crystal formation has ceased, cool the flask in an ice bath for 10 minutes to precipitate more crystals.

Collect the recrystallized solid by vacuum filtration using a Büchner funnel. Wash the crystals with a few mL of *ice-cold* water, and press them down with a spatula. Allow the vacuum to pull for at least 10 minutes to help dry the crystals. Scrape the crystals onto a pre-weighed beaker and determine the mass of the recrystallized unknown. Determine the melting point of the unknown and try to identify it from the table below. Conduct a mixed melting point experiment by melting a mixture of your unknown and the authentic sample you believe your solid unknown to be. If you observe a melting point depression of 5 ^{o}C or greater, repeat the mixed melting point experiment with the unknown with the next closest melting point.

Possible Recrystallization Unknowns

Compound	*mp (°C)*
o-toluic acid	103-105
m-toluic acid	108-112
benzoic acid	122-123
trans-cinnamic acid	131-136
m-nitrobenzoic acid	140-142
salicylic acid	158-161

Lab Report Guidance

Title and Introduction

- Provide a short title that clearly describes exactly what the experiment involves.
- What is the scientific purpose of this experiment?
- Provide a short summary of the experimental approach you will use.
- For each of the newly introduced methods used in this experiment (recrystallization, vacuum filtration, melting point, mixed melting point), describe (1) the principles or theory behind it and (2) its practical use.
- Include figures of any newly introduced equipment and apparatus used in this experiment.
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Experimental

Write the experimental section as shown below:

Unknown _____ (_____ g) was recrystallized from _____ mL of water to give _____ (color) _____ (form; i. e. crystals, powder, plates, etc.).

Mass: _____ g (_____ %) (the mass and % recovery of your product)
mp: _____ °C (give the accurate mp of your product here).

When mixed with an authentic sample of _____, no melting point depression was observed.

Results and Discussion

Provide and discuss the following:

Recrystallization Data

- Unknown number
- Initial mass of unknown
- Mass of recovered unknown
- Percent recovery
- ***Show percent recovery calculations:***

Melting Point Data

- Approximate melting point
- Accurate melting point
- Preliminary identification of unknown
- Mixed melting point with which compound(s) and the results of those experiments
- Final identification of unknown
- Provide a detailed explanation of 1) how you made your preliminary identification, and 2) how you confirmed that preliminary identification to make your final identification.

Conclusion

- Provide short paragraph (1 – 3 sentences) that summarizes what happened in the experiment, and whether or not your purpose was achieved.

Questions:

1. Why is the product collected after recrystallization washed with cold solvent rather than hot solvent?

2. Three test tubes, labeled A, B, and C, contain compounds with approximately the same melting point. Using only a melting point apparatus, how could you prove that the test tubes contain three different chemical compounds?

3. Table sugar (sucrose) is refined on a large scale by recrystallization. What volume of boiling water (in mL) is required to dissolve 100 g of sucrose? If the solution were then cooled to room temperature (~20 °C), how much sucrose (in g) would recrystallize out of the solution?