# 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 (Padias):**

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| **Topic** | **2nd Edition** | **3rd Edition** |
| Laboratory Notebook | pp. 5 – 13 | pp. 5 – 13 |
| Basic Lab Techniques | pp. 16 – 30 | pp. 16 – 30 |
| % Recovery Calculation | p. 15 | p. 15 |
| Solvents, Solubility | pp. 37 – 43 | pp. 37 – 43 |
| Melting Point | pp. 47 – 53 | pp. 47 – 53 |
| Recrystallization | pp. 119 – 127 | pp. 121 – 129 |

**Safety:**

Be certain to frequently stir or swirl any liquid while heating it to prevent “bumping.”

**Procedure:**

To a 50-mL Erlenmeyer flask add about 0.5 gram of a solid unknown. Add 10 mL of water and a boiling stick, then heat the flask until the contents boil. Be sure to swirl the mixture often while heating; when the mixture reaches boiling, swirl the hot mixture for about one minute. If necessary, add water to maintain volume during boiling. Continue to add water to the flask, 2-3 mL at a time, boiling for one minute after each addition until the unknown dissolves.

Once the unknown has dissolved, remove the flask from the heat, remove the boiling stick, and allow the solution 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. Place the product in a labelled, pre-weighed beaker and allow it to dry (uncovered) in the hood for at least one day. Reweigh the beaker to 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 oC or greater, repeat the mixed melting point experiment using the authentic sample with the next closest melting point.

**Possible Recrystallization Unknowns**

|  |  |
| --- | --- |
| Compound | *mp (oC)* |
| *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 |

Recrystallization Data

1. Unknown number: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

2. Initial mass of unknown: \_\_\_\_\_\_\_\_\_\_\_\_\_\_

3. Mass of recovered unknown: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

4. Percentage recovery: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ (show calculation)

Melting Point Data

5. Approximate melting point: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

6. Accurate melting point: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

7. Preliminary identification of unknown: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

8. Mixture melting point with:

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_, mp: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_, mp: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

9. Final identification of unknown: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

10. Explain how you made your preliminary identification of your unknown, and how you confirmed it.

**Questions**

**(Fully explain how you arrived at each answer, including all calculations.)**

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 30 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? Show all calculations.