Recrystallization and Melting Point Determination of Benzoic Acid

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Abstract: *[Write a concise summary of the experiment. Include: (1) the purpose—to purify impure benzoic acid via recrystallization and determine its melting point; (2) brief methods—dissolved in hot water, cooled for crystallization, vacuum filtered, measured melting points; (3) key results—report the melting points of impure and pure samples, and percent recovery; (4) conclusions—comment on the success of purification based on melting point data.]*

1. Introduction

Organic compounds are often impure, after synthesized in the laboratory or isolated from natural sources. This contamination needs to be removed before those compounds can be applied and utilized in other applications. One of the most common and important methods of purification, for an organic chemist to know, is **recrystallization**. It is essentially a technique to remove impurities from organic compounds that are solid at room temperature. Because the solubility of a compound in a solvent generally increases with temperature, if we allow the solution containing the compound to cool slowly until the solution becomes saturated, we can obtain relatively pure crystals because molecules in the crystals have a greater affinity for other molecules of the same type. Afterwards, the impurities are left in the solution, and the pure compound is isolated and crystallized.

The purpose of this experiment is to purify impure benzoic acid (C_6H_5COOH) that contains sugar, via recrystallization. Then, we will determine the melting point of both the impure and pure benzoic acid samples to confirm the success of the process of recrystallization and purification. Because the melting point of an impure compound—in this case, benzoic acid with sugar—will typically be a wider range at a higher temperature, and because the purified compound will have a much narrower range at a lower temperature, we can use the melting point to assess the purity of the compound.

Benzoic acid is a white or colorless crystalline organic compound whose structure consists of a benzene ring (C_6H_6) and a carboxyl substituent. The substance occurs naturally in many plants, and salts of benzoic acid are used as food preservatives. It is also an important element for the industrial synthesis of many other organic compounds.**[wikipedia_benzoic_acid]**

According to the PubChem Compound Summary for benzoic acid, the melting point of benzoic acid is around 122.4 °C and its solubility in water is around $3.4\,\frac{\text{mg}}{\text{mL}}$ at 25 °C **[pubchem_benzoic_acid]**. The melting point of sugar (sucrose) is around 185.5 °C and its solubility in water is around $2.12 \times 10^6\,\frac{\text{mg}}{\text{L}}$ at 25 °C **[pubchem_sucrose]**.

[Explain the theory behind recrystallization. Discuss how solubility changes with temperature and why impurities are left behind in solution.]

[Explain how melting point is used to assess purity. Discuss the relationship between impurities and melting point range/depression.]

[Provide background on benzoic acid (C_6H_5COOH), including its structure, physical properties, and solubility in water. Cite your sources.]

2. Experimental Details

2.1. Materials and Apparatus

- Impure benzoic acid sample (mass: 1.018 g)
- Deionized (DI) water
- 125 mL Erlenmeyer flask or beaker
- 150 mL beaker
- Hot plate
- Ice bath
- Vacuum filtration apparatus (Büchner funnel, filter flask, vacuum trap)
- Filter paper (mass: 1.099 g)
- Melting point apparatus
- Melting point capillary tubes
- Analytical balance
- Weighing boat

2.2. Procedure

2.2.1. *Day 1: Recrystallization*

1. Record the melting point of impure benzoic acid
2. Weigh out approximately 1.00 g of impure benzoic acid
3. Heat DI water to near boiling
4. Dissolve the impure benzoic acid in hot water (total volume added: 45 mL)
5. Allow solution to cool slowly to room temperature
6. Place in ice bath to enhance crystallization
7. Collect crystals by vacuum filtration
8. Allow crystals to dry overnight

2.2.2. *Day 2: Analysis*

1. Retrieve dried crystals from drying oven
2. Record mass of purified benzoic acid
3. Measure melting point of purified benzoic acid

3. Results

3.1. Experimental Data

Data is presented in Table 1 and Table 2.

Table 1: Summary of experimental mass and melting point data for benzoic acid recrystallization.

Measurement	Value	Unit
Mass of impure benzoic acid	*1.018*	g
Volume of hot deionizedwater used	*45*	mL
Mass of filter paper	*1.099*	g
Mass of filter paper + crystals	*X.XXX*	g
Mass of purified benzoic acid	*X.XXX*	g

Table 2: Melting point data for impure and purified benzoic acid samples.

Sample	MP Range	Width
Impure	*112–132* °C	*20* °C
Purified	*XX–XX* °C	*X.X* °C
Literature*	122.4 °C	—

*Literature melting point from the PubChem Compound Summary for benzoic acid.[pubchem_benzoic_acid]

3.2. Calculations

3.2.1. *Percent Recovery*

Before calculating the actual percent recovery from the results, we can first find the theoretical maximum of benzoic acid that can be recovered from the recrystallization. We can do this using the solubility of benzoic acid in water at 25 °C, which is around 3.4 $\frac{\text{mg}}{\text{mL}}$.

Thus,

$$\begin{aligned}
\text{Solubility} &= 3.4\,\frac{\text{mg}}{\text{mL}} \\
\text{Volume of water used} &= 45\,\text{mL} \\
\text{Maximum mass lost to solubility} &= 3.4\,\frac{\text{mg}}{\text{mL}} \times 45\,\text{mL} \\
&= 153\,\text{mg} \\
&= 0.153\,\text{g}
\end{aligned}$$

Therefore, the theoretical maximum recovery is:

$$\begin{aligned}
\text{Theoretical max} &= 1.018\,\text{g} - 0.153\,\text{g} \\
&= 0.865\,\text{g}
\end{aligned}$$

The percent recovery of benzoic acid was calculated using the following equation:

$$\%\ \text{Recovery} = \frac{m_{\text{pure}}}{m_{\text{impure}}} \times 100\% \tag{1}$$

(Show your calculation with actual values:)

$$\text{Percent Recovery} = \frac{\textit{mass recovered}\ \text{g}}{\textit{initial mass}\ \text{g}} \times 100\% = \textit{XX.X}\%$$

4. Discussion

4.1. Structure and Intermolecular Forces of Benzoic Acid

4.1.1. *Structure of Benzoic Acid*

[Draw the bond line structure of benzoic acid using ChemDraw or similar software, or use the chemfig package. Include the figure with a proper caption.]

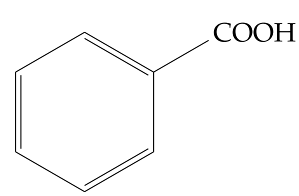

Figure 1: Bond line structure of benzoic acid (C_6H_5COOH).

4.1.2. *Intermolecular Forces in Benzoic Acid*

[Identify ALL types of intermolecular forces (IMFs) present in benzoic acid:

- *Hydrogen bonding (carboxylic acid group)*
- *Dipole-dipole interactions*
- *London dispersion forces*

Explain the origin of each type of IMF based on the molecular structure.]

4.1.3. Relationship Between IMFs and Melting Point

[Explain why benzoic acid has a relatively high melting point (122.4 °C) in terms of its intermolecular forces. Discuss how the strong hydrogen bonding between carboxylic acid groups, combined with the aromatic ring's dispersion forces, requires significant thermal energy to overcome.]

4.2. Analysis of Purification Results

4.2.1. Percent Recovery Analysis

[Discuss your calculated percent recovery from Section 1. Is this value reasonable? What factors affect percent recovery in recrystallization? Consider:

- *The solubility of benzoic acid in water at different temperatures*
- *The volume of water used*
- *Loss during transfer and filtration*

]

4.2.2. Comparison of Melting Points

[Compare the melting point of the impure benzoic acid to that of the purified sample (see Table 2). Address:

- *Was the melting point of the impure sample lower than the pure sample?*
- *Was the melting range of the impure sample wider?*
- *Do these observations make sense based on melting point depression theory?*

Explain the effect of impurities on the melting point of a substance.]

4.2.3. Purity Assessment

Write your purity assessment here based on the criteria above.

4.3. Error Analysis

Write your error analysis here, addressing at least 2–3 specific sources of error.

5. Conclusion

[Write a brief conclusion that:

- *Restates the purpose of the experiment*
- *Summarizes the key results (percent recovery, melting points)*
- *States whether the purification was successful based on melting point data*
- *Mentions any directions for improvement if the experiment were repeated*

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References