**RECRYSTALLIZATION**

From *Organic Chemistry with Vernier*

# EXPERIMENT 2 INTRODUCTION



**Westminster College**

A fundamental purification technique for organic solids is recrystallization, which uses the different solubility of solutes in a solvent. Generally, compounds isolated from organic reactions are impure and require purification to obtain the desired clean product. Recrystallization is a purification process because the slowly growing crystals incorporate only those same molecules that fit correctly into the crystal lattice. The impurities that do not fit into the crystal lattice remain in solution.

The solubility of the compound in the recrystallization solvent is important. In general, a minimal amount of hot solvent would completely dissolve the compound to be purified. Upon cooling, the impurities will remain in solution as the desired compound crystallizes. A suitable recrystallization solvent should also be somewhat volatile in order to be easily removed from the purified crystals.

If the cooling process is allowed to proceed slowly, nearly pure crystals of the compound will form. If the solution is cooled too quickly, the impurities will precipitate out of the solution along with the desired product. Once crystallized, the solid can be collected by vacuum filtration, washed with cold solvent, and dried. The purity of the recovered solid can be analyzed by determining the melting temperature.

# OBJECTIVES

In this experiment you will

* Recrystallize a sample of contaminated benzoic acid.
* Test the solubility of acetylsalicylic acid.
* Recrystallize acetylsalicylic acid from aspirin.
* Characterize the compounds by melting temperature analysis.

# MATERIALS

## Part I Recrystallization of Benzoic Acid

|  |  |
| --- | --- |
| 100 mL beaker | DI water |
| 250 mL beaker | Ice |
| Hot plate | Watch glass |
| Disposable Pasteur pipets and bulb | Compressed air |
| Glass stirring rod | Contaminated benzoic acid |
| Vacuum filtration apparatus |

## Part II Test the Solubility of Acetylsalicylic Acid

|  |  |
| --- | --- |
| Three 13 x 100 test tubes | Methanol, reagent grade |
| Test tube rack | Ethanol, reagent grade |
| 250 mL beaker | 2-propanol, reagent grade |
| Hot plate | Acetone |
| Spatula | DI water |
| Disposable Pasteur pipets and bulb | Ice |
| Glass stirring rod | Cold DI water in wash bottle |
| Wide-Range Temperature Probe or Thermometer | Acetylsalicylic acid |

**Part III Recrystallization of Aspirin**

|  |  |
| --- | --- |
| 250 mL beaker | Methanol, reagent grade |
| 125 mL Erlenmeyer | Ethanol, reagent grade |
| Hot plate | 2-propanol, reagent grade |
| Mortar and pestle | Acetone |
| Glass stirring rod | DI water |
| Disposable Pasteur pipets and bulb | Ice |
| Vacuum filtration apparatus | Cold DI water in wash bottle |
| Gravity filtration apparatus | Compressed air |
| Spatula | Watch glass |
| Wide-Range Temperature Probe or Thermometer | Aspirin tablets |

**Part IV Melting Temperature**

|  |  |
| --- | --- |
| LabQuest or computer interface | Benzoic acid |
| LabQuest App or Logger *Pro* | Acetylsalicylic acid |
| Vernier Melt Station | Samples from Parts I and III |
| Glass capillary tubes, one end closed | (optional) mortar and pestle |
| Tissues (preferably lint-free) |

**PROCEDURE**

**Part I Recrystallization of Benzoic Acid**

1. Obtain and wear goggles.
2. Weigh out 1.0 g of the contaminated benzoic acid sample. Record the mass to the nearest 0.001 g.
3. Transfer the solid into a 100 mL beaker. The solubility of benzoic acid in water is approximately 2 g/L at 25°C and 68 g/L at 95°C. Calculate the approximate amount of hot water needed to dissolve your sample of benzoic acid and add to the beaker.
4. Gently heat the solution using a low setting on the hot plate until the solid has completely dissolved. An additional 1–2 mL of water may be added if necessary.
5. While waiting for the solid to dissolve, prepare an ice water bath in a 250 mL beaker.
6. When the solid has completely dissolved, remove the flask from the hot plate and let it slowly cool to room temperature undisturbed. **Note:** If no crystal growth has occurred, scratch the inside bottom of the flask with a glass rod. If crystallization still

does not occur, reduce the volume of the solution by heating it again just until the solution turns cloudy or crystals appear. Add enough of your solvent to dissolve the crystals and let it cool as you did before.

1. After the solution has cooled to room temperature, further cool the flask in the ice water bath.
2. Collect the crystals using vacuum filtration. **Note:** Be sure to record the mass of the filter paper before placing it in the vacuum funnel.
3. Place the filter paper containing the filtered solid on a watch glass and gently direct a stream of air (low flow) to thoroughly dry the solid. When the solid has dried, weigh the recovered solid and record the mass to the nearest 0.001 g. Save the solid for the melting temperature analysis in Part IV.

## Part II Test the Solubility of Acetylsalicylic Acid

1. Prepare a 60–70°C hot water bath in a 250 mL beaker. Monitor the temperature of the water bath using a Wide-Range Temperature Probe or thermometer. **Note:** Save the hot water bath for Part III.
2. Obtain three 13100 test tubes and label then 1–3.
3. Prepare to test the solubility of acetylsalicylic acid.
	1. Select two solvents from the table below. Test Tubes 1 and 2 will contain the organic solvents. The third solvent will be water in Test Tube 3. Record this information in the data table.
	2. Put a small pea-sized amount of acetylsalicylic acid in the each of the test tubes.
	3. Use 1 mL of the organic solvents and 2 mL of water for testing.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Temperature (°C) | Methanol (g/L) | Ethanol (g/L) | 2-propanol (g/L) | Acetone (g/L) | Water (g/L) |
| 25 | 254.75 | 235.83 | 118.91 | 185.20 | 4.50 |

1. Observe the solubility of acetylsalicylic acid in your selected solvents at room temperature, in the hot water bath and in an ice water bath. Record your observations in your data table.

**Note:** When investigating the solubility in water, increase the temperature of the water bath to approximately 90°C.

1. Based on your observations, select the best solvent and conditions to recrystallize your aspirin sample for Part III.

## Part III Recrystallization of Aspirin

1. Obtain two aspirin tablets and crush the sample using a mortar and pestle.
2. Weigh and record the mass of the crushed aspirin tablets to the nearest 0.001 g.
3. Prepare the aspirin tablets for recrystallization.
	1. Transfer the sample to a 125 mL Erlenmeyer flask.
	2. Dissolve the solid in a minimal amount of your chosen solvent from Part II.
	3. Gently heat the solution in the hot water bath. An additional 1–2 mL of solvent may be added if necessary. **Note:** If using water as the recrystallization solvent, gently heat the flask directly on the hot plate.
4. Use gravity filtration to filter the hot solution into a clean Erlenmeyer flask to remove the insoluble impurities.
5. Let the solution slowly cool to room temperature then further cool the flask in an ice water bath. As the solution cools, the solid will begin to crystallize.
6. Collect the crystals using vacuum filtration. **Note:** Be sure to record the mass of the filter paper before placing it into the vacuum funnel.
7. Place the filter paper containing the solid on a watch glass and gently direct a stream of air (low flow) to thoroughly dry the solid. When the solid has dried, weigh the recovered solid and record the mass to the nearest 0.001 g. Save the solid for melting temperature analysis in Part IV.
8. Discard waste as directed by your instructor.

## Part IV Melting Temperature

1. Obtain a small amount of your sample from Part I. The solid should be in a powdered form. If it is not, use a mortar and pestle to carefully grind the solid to a powder. Pack a capillary tube 3–4 mm (~1/8 inch) deep with your sample.
2. Check the control dial on the Melt Station to confirm that it is in the Off position. Connect the Melt Station power supply to a powered electrical outlet.
3. Connect the Melt Station to a LabQuest or to a computer interface. Choose New from the File menu of the data-collection program.
4. Carefully insert the capillary tube of solid into one of the sample holders of the Melt Station.
5. Begin collecting melting temperature data using the Melt Station.
6. Adjust the control dial in order to determine the approximate melting temperature range for the sample.
7. When finished, stop data collection and turn the dial to the Fan/Cooling setting. Record the melting temperature range in your data table.
8. Store the run and collect a second run.
9. Repeat the necessary steps to collect melting temperature data for the sample from Part III.
10. At the end of the experiment turn the control dial on the Melt Station to Off. Dispose of the capillary tubes as directed by your instructor.
11. Complete the Data Analysis section before exiting Logger *Pro* or LabQuest App. Print a copy of your graph and/or save your data, as directed by your instructor.

# DATA TABLE

## Part I Recrystallization of Benzoic Acid

|  |  |
| --- | --- |
| Mass of sample (g) |  |
| Calculated volume of water needed (mL) |  |
| Mass of filter paper (g) |  |
| Mass of filter paper and product (g) |  |
| Mass of recrystallized product (g) |  |

**Part II Test the Solubility of Acetylsalicylic Acid**

|  |
| --- |
| Observations |
| Test tube | 1 | 2 | 3 |
| Solvent |  |  | water |
| Room temperature |  |  |  |
| Hot water bath |  |  |  |
| Ice water bath |  |  |  |

code: vs = very soluble, so = soluble, ss = slightly soluble, in = insoluble

Recrystallization solvent

## Part III Recrystallization of Aspirin

|  |  |
| --- | --- |
| Mass of aspirin sample (g) |  |
| Mass of filter paper (g) |  |
| Mass of filter paper and product (g) |  |
| Mass of recrystallized product (g) |  |

**Part IV Melting Temperature**

|  |  |
| --- | --- |
|  | Experimental melting temperature range (°C) |
| Benzoic acid |  |
| Aspirin |  |

**DATA ANALYSIS**

1. Calculate the percent recovery of benzoic acid and aspirin.
2. What are some of the properties of an ideal recrystallization solvent?
3. Why is ice cold solvent used to rinse the purified crystals?
4. If the hot solution were immediately placed in an ice water bath during the recrystallization process instead of first cooling to room temperature, how might this affect the results?