

# Solids: recrystallization and melting points essay sample

[Environment](#), [Water](#)



Abstract: The purpose of this lab was to purify an unknown compound by recrystallization. Taking an unknown compound and identifying it by purifying it from its impurities through the use of hot gravity filtration. Then to determine the unknown you were to take the melting point. An unknown compound was recrystallized from hot water to produce 0.99 grams (67% recovery). The pure unknown compound had a melting point of 127.0-128.9°C. The mixed melting point of the unknown with the benzoic acid was 107.9-109.1°C and with the 2-ethoxybenzamide was 127.5-128.4°C so the compound must be 2-ethoxybenzamide.

Introduction: This experiment was conducted in order to explore the methods of recrystallization and in order to determine the melting points of various solids. Recrystallization is to purify a solid by allowing it to recrystallize out the solution. The impurities stay in solution while the desired solid precipitates out. You have to dissolve a solute in a hot solvent and, then recrystallize the solute out of the cooled solvent. The formation of concentrated, pure crystals is possible in this recrystallization process if the appropriate cooling rate is applied to the solute and solvent mixture.

Consequently, a purified, solid compound would be produced.

Recrystallization involves dissolution of a solid in a solvent at elevated temperatures and the reformation of the crystals as the solution cools, allowing for impurities to remain in the solution. Once a solid has been recrystallized, it is important to determine the purity of the recrystallized solid.

Experimental Procedure:

**Cautious:**

- Vapors generated in this experiment are nasty. Carry out this experiment in the hood. Use caution when working with hot plates and hot solutions: it is easy to burn oneself when pouring boiling water or if one's solution bumps.

**Procedure:****Part A:**

Dissolve the compound in a minimum of boiling water.

Skip the gravity filtration step (no insoluble impurities are present). Allow the solution to slowly cool to room temperature. (At least 20mins) Cool the solution in ice for 20 minutes.

Collect the crystals by vacuum filtration.

Place your crystals in an oven at 90 degree Celsius on a watch glass to dry for at least 20 minutes. Part B:

Repeat on a 2nd sample.

Repeat the procedure above

Dissolve the compound in 100mL of water.

Dissolve the compound in a minimum boiling water and then boil the solution long enough to reduce its original volume to half. Allow the solution to cool to room temperature for only one minute and then cool in ice for 20 minutes. Allow your second set of crystals to dry in an oven for 90 degree Celsius for at least 20 minutes. Determine the melting point of the original impure sample.

Determine the melting point and yield of both sets of purified crystals.

Report your results in tubular form.

Submit both sets of crystals in labeled vials.

Results:

Solvent

Physical State

Molecular Weight (g/mol)

Melting Point (C)

Benzoic Acid Unknown 9B

Liquid

34.08g

126C

Benzoic Acid Unknown 1B

Liquid

24.43g

125C

The table explains my two benzoic acid of unknowns 9B and 1B. First, take the plate and weighed it as 32.902g, the paper was .182g and 1.00g of acid was added to the total of 34.084g. By taking the benzoic acid with the instructions to prepare the unknown for the moderate amount of H<sub>2</sub>O (20mL/10mL) slowly. After it iced for 20 minutes the crystals were ready to collect in the filtration. Then, it was placed in the oven for at least 20 minutes, and then got its weight and the crystals were ready to be placed in

the digital scales to be placed in the dig melt to find its melting point, which was 126C.

With the second sample of benzoic acid 1B the plate weighed at 23. 260g, the paper was . 170g and 1. 00g of acid was added and the total weighed out to be 24. 43g. Using this sample the guidelines were to perform a moderate amount of H<sub>2</sub>O (20mL/10mL) quickly. After it iced for 20 minutes the crystals were ready to collect in the filtration. Then, it was placed in the oven for at least 20 minutes, and then got its weight and the crystals were ready to be placed in the digital scales to be placed in the dig melt to find its melting point, which was 125C.

#### Discussion:

During the cold filtration, the water soluble impurities that might dissolve in water which was filtered out through the suction filtration. However, some of the impurities might be trapped on the surface of the benzoic acid crystals, so a small volume of ice-cold water should be used to wash the benzoic acid crystals to dissolve the particular impurities. The crystal was dried in the oven at 100 °C. A fresh piece of filter paper can be used to place under the filter paper with benzoic acid crystals. Benzoic Acid unknown 9B melting point range from 116C-126C and the unknown benzoic acid of 1B ranged from 122. 3C-125C.

The purity of a crystal can be determined by its melting point. A narrow range of melting point indicates high purity of the sample, otherwise broad range of melting point indicates the presence of impurities in the crystal. The

melting point of the recovered benzoic acid obtained experimentally is 126 °C. Compared to the pure benzoic acid with 125°C of melting point, the purity of the recovered benzoic acid is very high with 98.36%.

Questions:

1.

Tabulate the part B results you obtained and those of at least three others who carried out different modifications.

2a

Discuss how the following factors affected the yield and purity: quantity of solvent rate of cooling

-This happens mostly because it is not dry, if there are still extraneous solvents when working with micro scale reactions, it can really mess up your yields. -Also reading the graduated cylinder correct.

2b

In each case, explain why the yield and purity were improved or worsened. -

Dissolve the compound in a minimum of boiling water and then boil the solution long enough to reduce its original volume to half. -Allow the solution to cool to room temperature for only one minute and then cool on ice for 20 minutes.