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Organic Lab 309:03

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Experiment 3: Recrystallization and Melting Point Measurement

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Purpose: The purpose of this experiment is to purify and identify the unknown component of Panacetin.

Equations:

Mechanisms:

Amounts and Properties:
Table 1: Chemicals and the Properties

Chemicals	Molar Mass (g)	Melting Point	Solubility c.w	Solubility b.w
Acetanilide	135.2	114	.54	5.0
Phenacetin	179.2	135	.076	1.22

Hazards and Safety: Acetanilide and Phenacetin can irritate skin and eyes so wear gloves and goggles at all times. To dispose, put the waste down the respective labeled containers.

Procedure:
Purification:

1. Recrystallize unknown component by boiling just enough water to dissolve it completely and then let it cool slowly to room temperature.
2. Get a large Erlenmeyer flask that can hold the largest volume of recrystallization solvent that was calculated. If necessary, induce crystallization by scratching sides of flask with the stirring rod.
3. Cool container further in a beaker of ice and tap water.
4. Collect solid by vacuum filtration and wash with small amount of ice water.
5. Dry product to constant mass and weigh in tared vial.

Analysis:

1. Grind a small amount of dry component to a fine powder on a watch glass. Divide the powder into 4 equal portions.
2. Mix pile 1 and pile 2 together. Mix pile 3 with an equal amount of acetanilide and mix pile 4 with an equal amount of phenacetin.
3. Measure the melting point of pile 1 and 2, acetanilide mix and phenacetin mix.
4. Record the temperatures where there is the first trace of liquid and where the sample is completely liquid.
5. Turn in remaining product in a vial labeled with the actual product name given.

Observations:

During the reboiling process, the water turned into a cloudy solution that took a while to dissolve all the unknown component. The calculated volume that was required to dissolve the unknown component was less than that of the actual volume of water used. As the majority of the component was dissolved, during the cooling process, the crystals immediately formed and had a bright shine to them. During the filtration, the solid looked like the solid from Experiment 2, which meant that there were little to no impurities. Within the melting point determination, there as soon as the three samples began to melt, the samples became liquids that were murky white in color.

Measurements:

Table 2: Melting Point Measurements

MIXTURES	Initial Liquid Spot Temp	Fully Liquid Temp
Mix 1 and 2	125°C	131°C
Acetanilide Mix	110°C	114°C
Phenacetin Mix	125°C	131°C

Data and Calculations:

Table 3: Experimental Data During Lab

Acetanilide Volume: 22.56mL	Phenacetin Volume: 92.459mL
Mass Recovered: .589g	Total Volume Used: 132.459mL

Acetanilide Volume: $1.128\,\frac{g*100\,mL}{5\,g}=22.56\,mL$

Phenacetin Volume: $1.128\,\frac{g*100\,mL}{5\,g}=92.459\,mL$

Total Volume: 92.459mL + 40.0mL = 132.459mL

Discussion:

From experiment 2, the recovered amount of the unknown was 1.128g which lead to the calculations of 22.56mL and 92.459mL for Acetanilide and Phenacetin respectively. During the experiment, the sample that was acquired had a hard time dissolving in boiling water which required a higher amount of water to be used than that of the calculated value. The calculated value was 92.459mL for Phenacetin and there was a need for an extra 40mL to be added so that the sample could finally dissolve. This lead to the thought of the unknown being Phenacetin because of the amount of water that was required to dissolve the large sample. From the melting point segment of the experiment, The mix that had portion 1 and 2 began melting at 125°C but

fully melted at 131°C. The mix with acetanilide began melting at 110°C and fully melted at 114°C. The mix with phenacetin began at 125°C and melted at 131°C. Comparing the mix's melting points, the unknown component was thought to be as Phenacetin because of the similar melting points with the third mix.

Conclusions:

Since the amount of water that was required to dissolve the unknown was greater than the calculated amount for Phenacetin and the melting point of the sample mixture was the same as the Phenacetin sample mix, the unknown component from Unknown #2 was Phenacetin.

Exercises:

1. A. $1.15\,\frac{g*100\,mL}{1.22\,g}=94.262\,mL$

   B. $94.262\,\frac{mL*.076\,g}{100\,mL}=.0716\,g$

   C. $1.15\,g-.0716\,g=1.0784\,g$
2. Compound X is phenyl succinate. Since both benzoic acid and m-aminophenol lower the boiling point being 89 and 102 respectively, when phenyl succinate is added together with the unknown it is about 120 which is close to the 121 degrees C melting point.
3. A. If the product is not dry, the scale will record a higher mass which increases the yield. This is because of the water that is excess with the component that is tested for.

   B. Yield is lowered due to the amount of water used since acetanilide has a higher solubility than that of phenacetin. During the drying process the components wouldn't be able to fully crystallize with that much water.
4. For the first scenario, the melting point would be messed up considering the water would have to evaporate before accurate data can be measured. For the second scenario there should be no effect on the melting point.