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Organic Lab 309:03

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Experiment 2: Separating the Components of “Panacetin”

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Purpose: The purpose of this lab is to determine the contents of Panacetin after separating each component.

Equations:

Mechanisms:

Amounts and Properties:
Table 1: Amounts and Properties of chemicals used in Lab

Chemicals	Mol. Mass (g/mol)	Amt (g, mg, or mL)	Actual Amt (g, mg or mL)	mmol	Mb/bp	Density (g/mL)	Theoretical Yield
Panacetin		3.00g					
Dichloromethane	84.927	50mL					
5% Sodium Bicarbonate	84.007	60mL, 2 30mL portions					
6M Hydrochloric	36.46	7.0mL					
Sucrose	342.3						8-12%
Aspirin	180.158						35-45%
Unknown							45-55%

Hazards and Safety: Avoid ingesting, inhaling, or skin contact with dichloromethane. To dispose of dichloromethane, dispose of it within the designated chlorinated solvent recovery container.

Procedure:

Separation of Sucrose:

1. Weigh 3.00g of Panacetin into a dry 125mL Erlenmeyer flask.
2. Add 50 mL of dichloromethane to flask and stir to dissolve as much as possible.
3. Weigh a fluted filter paper and filter the mixture by gravity into a small flask while keeping the filtrate (liquid that went through the filter paper).
4. Keep filter paper and put it aside and re-weigh the filter paper when completely dry.
5. Record mass of sucrose in the lab notebook and if requested, submit sucrose to instructor in a tared and labeled vial.

Separation of Aspirin:

1. Take filtrate and transfer into a separatory funnel and extract it with 2 separate 30mL portions of 5% sodium bicarbonate.

2. For each extraction, stir liquid layers until any fizzing subsides before stoppering and shaking the separatory funnel.
3. Transfer each layer to different containers and label them. Dichloromethane is on the bottom.
4. Return the dichloromethane layer back to the funnel before the second extraction.
5. Combine both two aqueous extracts int the same container and save the dichloromethane layer for the next part.
6. Slowly add 7.0mL of 6M hydrochloric to the combined aqueous extracts while stirring.
7. Test pH of solution and add more acid to bring pH to 2 or lower if needed.
8. Cool mixture in ice/water bath for at least 10 minutes, collect the aspirin by vacuum filtration, and wash it on the filter with cold water.
9. Let aspirin dry on filter for a few minutes with the aspirator running then dry it to constant mass.
10. Weigh aspirin and record its mass in lab notebook and submit to instructor in a vial.

Isolation of the Unknown Component:

1. Use filter flask attached to a trap and aspirator to evaporate the solvent from the dichloromethane solution.
2. Heating and swirling solution over a steam bath or in a hot water bath increases evaporation rate.
3. Stop evaporation when only a solid residue remains in flask or when no more solvent evaporates. Transfer unknown component to a tared vial and let it dry to be weighed.
4. Calculate percent recovery, dividing sum of the masses of all components by the mass of Panacetin that you started with. Calculate approximate percentage composition of Panacetin based on total mass of components recovered.

Observations:

The unknown sample that was acquired was vial #2. When the dichloromethane was added to the sample, there was no color change. When transferring everything into the separatory funnel, 5% $NaHCO_3$ was added that created fizzing which was probably CO_2 gas from the reaction in the funnel. After mixing the combined sample around, the top layer was a cloudy clear layer and the bottom was completely clear. After acquiring the filtrate, HCl was added that created a foamy, bubbly solution. While heating the sample was a whitish color. Sucrose after recovery was a small white powder portion and Aspirin was a large white shining pile.

Measurements:

Table 2: Measurements during the Lab

Panacetin Weight	~3.00g

Mass of Round Boiling Flask	95.656g
Mass of Round Boiling Flask and Unknown	96.784g

Data and Calculations:

Table 3: Calculated Data

Aspirin: .829g	35.92% Aspirin
Sucrose: .351g	15.208% Sucrose
Unknown: 1.128g	48.87% Unknown
2.308g Recovered	76.93% Recovery

Total Recovered: .829g + .351g + 1.128g = 2.308g

Total Recovery: $\frac{2.308\,g}{3.00\,g} * 100 = 76.93\,\%$

Aspirin %: $\left(\frac{.829\,g}{2.308\,g}\right) * 100 = 35.95\,\%$

Sucrose %: $\frac{.351\,g}{2.308\,g} * 100 = 15.208\,\%$

Unknown: 96.784g - 95.656g = 1.128g

Unknown %: $\frac{1.128\,g}{2.308\,g} * 100 = 48.87\,\%$

Discussion:

At the beginning of the experiment, trying to weigh out the 3.0 grams of the Panacetin, the scale was under a lot of wind which lead to the scale stuttering between -.05g and .05g. The total recovery of the sample was 2.308g out of 3.0g which equals a 76.93% recovery rate. This means that some of the sample could've been lost within the filtering step and during the unknown filtering. From the textbook, a range of 8%-12% for sucrose is decent enough, 35-45% for aspirin, and 45-55% of the unknown. From the experiment the values from this trial was 15.208%, 35.95% and 48.87% respectively. These values were around the "real" labeled value which means this test was relatively accurate.

Conclusions:

Since the results showed the percentages of each component to be around the labeled value, this experiment proved that the labels on Panacetin is accurate.

Exercises:

1. When 6M HCl was added to Sodium Acetylsalicylate, there was fizzing that occurred which created foamy bubbly like substances on the surface of the sample. This changes occured because when HCl reacts with Sodium Acetylsalicylate, the Sodium attached to the Oxygen can be attacked by the Hydrogen from HCl creating aspirin.
2. A. Not mixing the dichloromethane and Panacetin long enough leads to some components not being able to be filtered out because the components won't be able to dissolve fully. This causes a lowered amount of component recovery.
   B. Not mixing the organic and aqueous layers thoroughly leads to a lower yield from the extraction since some components may be in the unwanted layer.
   C. Mixing the solution with HCl will make aspirin to form into Acetylsalicylate which is not the component that is tested for. This leads to a lowered yield of aspirin from the experiment.
   D. Since the pH has to be low for the reaction to occur, if the pH goes back up to 7 the reaction can no longer go forward and the yield would be lower.
3. When acetanilide and acetaminophen are extracted with the 5% NaOH they are able to create water soluble compounds.
   a. (Aspirin)COOH + NaOH → (Aspirin)COO- Na+
   b. (Acetaminophen)COOH + NaOH → (Acetaminophen)COO- Na+
4. S

5. A. $Aspirin + NaHCO_3 \rightarrow AcetylsalicylateNa + H_2CO_3$

Aspirin is stronger acid, NaHCO3 is stronger base, AspirinNa is weaker base and H2CO3 is weaker acid

B. $AcetylsalicylateNa + HCl \rightarrow Aspirin + H_2O$

Sodium Acetylsalicylate is stronger base, HCl is stronger acid, Aspirin is weaker acid and water is weaker base