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Letters

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EXP1: Extraction of 3 component mix

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Introduction:

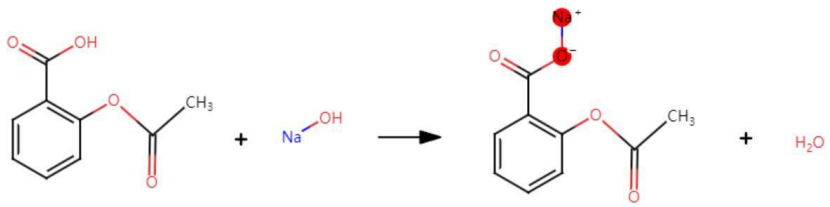
In this experiment, we employed acid-base extraction to isolate individual compounds from a mixture. This technique is widely used in chemistry and biology due to its efficiency in separating compounds based on their solubility in acidic or basic environments. A recent study, titled "Enhancement of β-Glucan Biological Activity Using a Modified Acid-Base Extraction Method from Saccharomyces cerevisiae" demonstrated an improved acid-base extraction approach that enhances the effectiveness of beta-glucan extraction, illustrating the practical applications of this method in biochemistry.

Acid-base extraction operates by adjusting the pH of a solution, thereby altering the solubility of different components and causing them to separate into distinct layers. In our experiment, we aimed to extract acetanilide, aspirin, and urea from a mixture containing these compounds. Initially, the mixture was combined with dichloromethane (DCM) to dissolve some of the compounds, while the undissolved substances were filtered out. Then, by adding sodium hydroxide (NaOH), we induced layer formation in the solution, allowing for the targeted extraction of compounds from each separated layer.

Results:

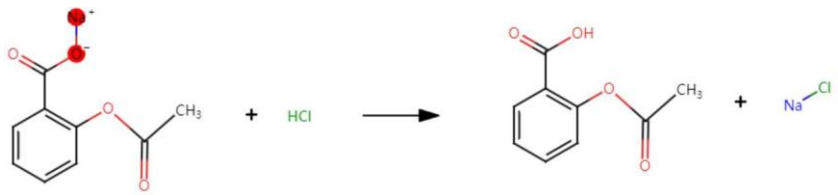
By dissolving the mixture with DCM, we can filter out the Urea that is not dissolved by DCM. By dissolving the mixture with DCM, we can filter out the Urea that is not dissolved by DCM. Through the experiment, we obtained 0.67g Urea. The theoretical yield is 1g, so the recovery rate of this compound is 67%.

Adding sodium hydroxide, the aspirin is extracted into the water layer in the form of sodium salt:



At this point, the Aspirin is in the aqueous layer, acetanilide remains in the organic layer.

Add magnesium sulfate to the separated organic layer and allow it to dry. The solution was then evaporated with a heating plate. After the liquid was completely evaporated, 0.85g acetanilide was obtained, so the recovery rate of acetanilide was 85%.



Acidifying the water layer with hydrochloric acid (HCl) regenerates the aspirin and is filtered and collected. For this experiment, we collected 0. 81g of aspirin, so the recovery rate of aspirin was 81%.

Discussion:

In this experiment, three compounds—acetanilide, aspirin, and urea—were recovered, with an average recovery rate of approximately 77.7%. The layer positions in the separatory funnel are based on density; typically, organic solvents, which are denser than water, form the bottom layer, while the aqueous layer is positioned on top.Several factors contributed to the low and variable recovery rates observed in this experiment. The main reason for the reduced yield is likely the incomplete separation of the aqueous and organic layers, leading to a partial loss of the target compounds. Additionally, product loss may have occurred due to improper transfer techniques. To minimize product loss, it's essential to handle the transfer carefully and ensure each container is thoroughly rinsed with solvent to capture any remaining material.

To enhance the recovery rate, one could allow more time for complete layer separation and, if needed, adjust the vacuum pressure to improve the efficiency of filtration. These adjustments could help achieve a more stable and higher recovery rate by reducing product loss and ensuring a more thorough separation.

Conclusions:

In this experiment, we used acid-base extraction to separate three distinct compounds from a mixture. By taking advantage of each compound's solubility in specific solvents and its acidity, we stratified the solution into different layers. This approach allowed us to isolate and collect each compound individually, resulting in the successful extraction of three separate compounds from the original mixture.

References:

(1) Mahmoud Amer, E.; Saber, S. H.; Abo Markeb, A.; Elkhawaga, A. A.; Mekhemer, I. M. A.; Zohri, A.-N. A.; Abujamel, T. S.; Harakeh, S.; Abd-Allah, E. A. Enhancement of β-Glucan Biological Activity Using a Modified Acid-Base Extraction Method from Saccharomyces Cerevisiae. *Molecules* **2021**, *26* (8), 2113. https://doi.org/10.3390/molecules26082113.

Supporting Information for "EXP1: Extraction of 3 component mix"

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Experimental Procedures:

1. Add 50 mL of dichloromethane (DCM) to the conical flask containing the mixture, and gently swirl for about 1 minute to dissolve the compound.
2. Prepare a vacuum filter and pour the solution from the conical flask through the vacuum filter and filter funnel to isolate compound A.
3. Use the remaining cold DCM to rinse the residue in the conical flask, ensuring as much compound A as possible is transferred to the funnel.
4. Use a vacuum pump to dry the solid on the filter paper for about 2 minutes. Transfer the solid to weighing paper, weigh it, calculate the recovery rate, and store it in a plastic bag for safekeeping.
5. Transfer the filtrate from the filtration step into a 250 mL separatory funnel. Rinse the vacuum flask used for filtration with 5 mL of DCM and add the rinse solution to the separatory funnel to ensure complete transfer of all compounds.
6. Add 25 mL of 1M NaOH (sodium hydroxide) to the separatory funnel, cover with the stopper, and shake well.
7. Allow the layers to separate completely. The organic layer will be on the bottom, and the aqueous layer on top. Separate the aqueous layer into a beaker labeled "Compound C."
8. Add another 25 mL of 1M NaOH to the separatory funnel and separate the aqueous layer again. Pour the organic layer into a beaker labeled "Compound B."
9. Add 1-2 small scoops of anhydrous magnesium sulfate to the beaker containing the organic layer. Transfer the dried organic layer into a 125 mL conical flask, leaving the magnesium sulfate residue behind.
10. Use a heating plate to evaporate the solution and collect compound B.
11. Add 10 mL of HCl to the beaker containing the aqueous layer and test the solution's acidity.
12. Cool the acidified solution in an ice bath for 5 minutes, then use vacuum filtration to separate the precipitate. Weigh the dried solid C, record its mass, and calculate the recovery rate.

Calculations:

$$\text{Percent Yield} = \left(\frac{\text{Actual Yield(g)}}{\text{Theoretical Yield(g)}}\right) \times 100$$

$$\text{Compound A} = 0.67\text{g Percent Yield} = \frac{0.67}{1} \times 100$$

$$\text{Compound B} = 0.86\text{g Percent Yield} = \frac{0.85}{1} \times 100$$

$$\text{Compound C} = 0.82\text{g Percent Yield} = \frac{0.81}{1} \times 100$$