Lab 6: Separating and Identifying the Components of a Mixture

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Objectives

By the end of this laboratory, you should have developed the skills to do the following:

- Separate components of a mixture using the principles of solubility and acid/base chemistry.
- Identify an unknown component based on its melting point.

Scenario

"Headache Gone" is an analgesic drug preparation commonly used for headaches that supposedly consists of 10% sucrose, 40% aspirin, and 50% acetaminophen. Your lab received several samples of Headache Gone for analysis. The analytical department has already analyzed the samples by HPLC (high-performance liquid chromatography) and determined that the samples do not contain acetaminophen as stated on the label, but instead contain a third, unknown component. They have narrowed down the identity of the unknown component to be either acetanilide or phenacetin. The samples have been sent to your department for identification of the unknown component and to determine the percent composition of each component. In order to do this, you will have to separate out the three components of the mixture using the principles of solubility and acid/base chemistry.

Background

In this lab, you will use the skills you have learned in previous labs (i.e., melting point, extraction, and recrystallization) to separate a mixture of three components and determine the percent composition of the mixture. The mixture contains two known components (sucrose and aspirin) and one unknown component. Once you have separated the sucrose and aspirin from the unknown, you will recrystallize your unknown to purify it (fortunately, both acetanilide and phenacetin can be recrystallized from water) and then take a melting point.

Percent composition is determined by dividing the mass of a specific component by the total mass of the sample and then multiplying by 100%. Ideally, the percent composition of the three components will add up to 100%, however it is likely that loss of material over the course of the lab will result in a total of slightly less than 100%. Make sure that all of your components are completely dry before weighing them to determine percent composition. A sample calculation for sucrose is shown below (for a student who started out with 3.050 g of the initial Headache Gone mixture and isolated 255 mg of sucrose). This is relatively close (within 2%) of the expected 10% composition, so in this example, we can consider the percent composition of sucrose to be "correct".

$$\text{Percent composition of sucrose} = \frac{\text{mass of sucrose isolated}}{\text{total mass of sample}} \times 100\% = \frac{0.255\ \text{g}}{3.050\ \text{g}} \times 100\% = 8.36\%$$

The identity of the unknown is determined primarily based on melting point, although solubility in hot water is an excellent indicator as well. When you are conducting the recrystallization, note how much hot water is needed. Phenacetin is significantly less soluble in hot water (1.22 grams per 100 mL of boiling water) than acetanilide is (5.0 grams per 100 mL of boiling water). Further, if you have successfully purified your unknown by recrystallization (as indicated by a narrow melting point range), you should easily be able to tell the difference between phenacetin (135 °C) and acetanilide (114 °C). As further confirmation, you can mix some of your sample with phenacetin and/or acetanilide and perform a mixed melting point. If your unknown is mixed with an impurity, the melting range will be significantly different and broader. If your unknown is purer, the melting range should be about the same.

Lab Notebook Preparation

Before coming to lab, the following items must be in your lab notebook:

1. Title of experiment & date the experiment is to be performed
2. Structures of sucrose, aspirin, acetanilide, and phenacetin
3. Melting points for acetanilide and phenacetin
4. Water solubility for acetanilide and phenacetin in boiling water
5. Reaction for the conversion of aspirin to sodium acetylsalicylate
6. References

Directions

1. Weigh approximately 3 g of "Headache Gone" and transfer it to a 125 mL or 250 mL Erlenmeyer flask. Be sure to record the exact amount of Headache Gone that you transferred to the flask.
2. Add 50 mL of dichloromethane to the flask.
3. Stir the mixture thoroughly to dissolve as much solid as possible (use your stirring rod to break up any lumps or granules).
4. Use gravity filtration to separate out the solid that did not dissolve. (What is the identity of the solid material? You will have to analyze the structures of the possible components of the mixture to determine which is least soluble in dichloromethane.)
5. Rinse the solid with a few milliliters of dichloromethane.
6. Label the solid and leave it to dry in your drawer until the next lab period.
7. Transfer the dichloromethane from steps 4 and 5 to a separatory funnel.
8. Wash the dichloromethane with 25 mL of aqueous 1 M sodium hydroxide or 5% sodium bicarbonate.
    a. ***Your instructor will assign the base you will use for your extraction.***
    b. Add 25 mL of the assigned base to the dichloromethane in the separatory funnel.
    c. Stopper the separatory funnel and shake it gently, venting frequently.
    d. Let the mixture sit in the ring clamp until the two layers have fully separated. Note which layer is on top and which layer is on the bottom.

   d. Remove the aqueous layer and retain it for future use. (What component are you removing from the dichloromethane? What component remains in the dichloromethane? You will have to analyze the structures of the possible components of the mixture to determine which has an acidic proton that can be removed by sodium hydroxide and how that might affect its solubility.)

9. Wash the dichloromethane a second time with 25 mL of your assigned base.
10. Label the container containing the dichloromethane and leave it uncovered to dry in your drawer until the next lab period. The dichloromethane should evaporate off to give you a solid material. If it does not, you can remove any remaining solvent under vacuum.
11. Combine the aqueous washes from steps 8 and 9 (you should have 50 mL total).
12. While stirring, slowly add 10 mL of 6 M hydrochloric acid to the aqueous layer (What reaction is occurring?).
13. Use a clean pipet to transfer a drop of the solution (attempt to avoid any solid that may have formed) to a strip of pH paper. Verify that the pH of the solution is 2 or lower. If it is not, add additional 6 M HCl until you have reached a pH of at least 2. Be sure to record how much additional HCl was needed.
14. Cool the solution to 10 °C or below by setting the beaker in a larger beaker containing ice and water.
15. Separate the solid from the solution using vacuum filtration. (What is the identity of the solid material?)
16. Label the solid and leave it to dry in your drawer until the next lab period.
17. At this point, you should have three separate solid materials (one from step 6, one from step 10, and one from step 16) that you have identified as sucrose, aspirin, and the unknown. Note their appearance (i.e., color and quality of crystals).
18. Recrystallize the crude aspirin to obtain pure product. Use ethanol and water as your recrystallization solvent. *NOTE: This process will be fast, so be prepared!*
    a. Begin by heating a beaker of water on a hotplate. Transfer your crude aspirin to a beaker and add approximately 10 mL of ethanol.
    b. When the beaker of water has reached a low, rolling boil, place your beaker of aspirin and ethanol on the hot plate.
    c. As the beaker of aspirin and ethanol come to a boil, you will see the solution turn clear. Once clear, begin adding 1 mL of hot water to your crude aspirin.
    d. The amount of hot water varies, but once the solution turns cloudy, immediately take off hot plate.
    e. Let purified crystals cool to room temperature on the benchtop, then place in an ice bath.
    f. Use a vacuum filter to separate your purified crystals.
    g. Place in lab drawer to dry.
19. Weigh the dry sucrose and the dry aspirin.

20. Note the appearance of the product (i.e., color and quality of crystals).
21. Measure the melting point of the dry aspirin.
22. Recrystallize the crude unknown to obtain pure product.
    a. Recrystallize the unknown. Use water only as your recrystallization solvent. Be sure to note how much water is used as this information will support the identity of your unknown.
23. Collect the pure crystals via vacuum filtration.
24. Leave the crystals to dry in your drawer until the next lab period.
25. Note the appearance of the product (i.e., color and quality of crystals).
26. Measure the melting point of the dry unknown product.
27. Take a mixed melting point with the substance you believe to be your unknown.
28. Weigh the dry product.
29. Determine the % composition and the % yield of your separation.
30. Record your final conclusions.

Reporting your Results

Write your report according to the guidelines described in "Topic 4: Writing an Organic Chemistry Lab Report". Work by yourself on this report.

References & Additional Resources

1. Lehman, J. W. *Operational Organic Chemistry: A Problem-Solving Approach to the Laboratory Course*, 3rd ed.; Prentice Hall: Upper Saddle River, NJ, 1999; pp 27-37.