Lab 5: The Effect of pH on Sodium Benzoate

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Objectives

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

- Separate a solid product from a reaction mixture via vacuum filtration.
- Purify a solid compound via recrystallization.

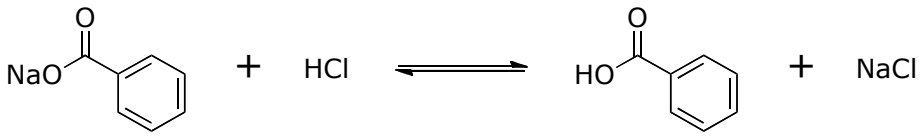
Recommended Resources

- Handout ~ Topic 4: Writing an Organic Chemistry Lab Report
  http://www.csub.edu/chemistry/organic/manual/Topic4_Report.pdf
- Handout ~ Topic 6: Common Lab Calculations
  http://www.csub.edu/chemistry/organic/manual/Topic6_Calculations.pdf
- Tutorial ~ Introduction to Crystallization Tutorial
  https://www.youtube.com/playlist?list=PLC37691582723F6F2
- Video ~ Recrystallization DanceChemistry
  https://youtu.be/jU-H4kidvXQ
- Handout ~ Professor David Evans's pKa table
  http://www2.lsdiv.harvard.edu/pdf/evans_pKa_table.pdf

Background

Recrystallization is a method frequently used to purify compounds that are solids at room temperature. In this process, a solvent (or mixture of solvents) is identified in which the substance to be purified is more soluble at high temperatures than it is at lower temperatures. The crude, or unpurified, compound is then heated with a small amount of solvent. This amount of solvent should be just enough to completely dissolve the solid while hot, but not enough to dissolve it while cold. If any solid impurities are present, they can be filtered out while the solution is hot using hot gravity filtration. As the solution cools, the pure solid will precipitate or crystallize out of solution while the impurities will remain in the solution. In this lab, you will perform an acid/base reaction and then purify the resulting product via recrystallization.

Sodium benzoate is a chemical commonly used as a food preservative. If sodium benzoate becomes protonated, it will form benzoic acid. In your prelab work, you will look up the pK_a of HCl and the pK_a of benzoic acid to determine if HCl is a strong enough acid to convert sodium benzoate to benzoic acid. The chemical equilibrium shown on the next page depicts the reaction that you will be investigating. Your experiment will determine whether the equilibrium lies to the left or to the right. Be sure to clearly indicate which side of the chemical equilibrium is favored both in your lab notebook and in your lab report



sodium benzoate benzoic acid

Benzoic acid is much less soluble in room temperature water than sodium benzoate, so if any benzoic acid is formed, it will precipitate out of solution. Your results will allow you to develop a prediction as to what might happen to sodium benzoate in the acidic environment of the stomach once it has been ingested.

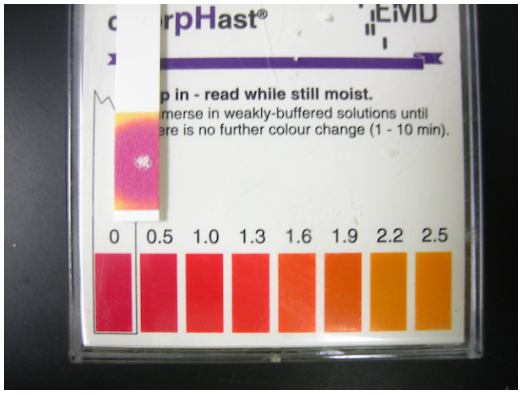
Lab Notebook Preparation

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

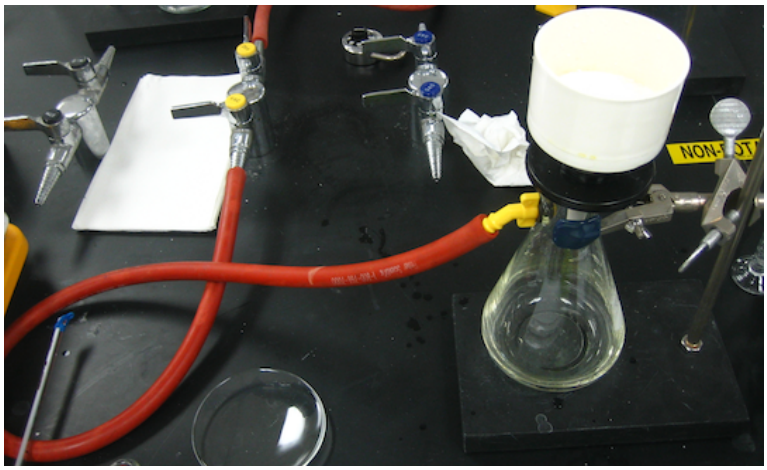
1. Title of experiment
2. Date the experiment is to be performed
3. The chemical reaction you are attempting (with skeletal structures)
4. A table with information about your starting materials. Include molecular weight, molar equivalents, and mmoles to be used. For solids (i.e., sodium benzoate) include grams to be used. For liquids (i.e., HCl), include the concentration.
5. The molecular weight and theoretical yield of the product (i.e., benzoic acid)
6. Any relevant physical properties (i.e., melting points of sodium benzoate and benzoic acid)
7. Hazards of and appropriate precautions for the safe handling of hydrochloric acid
8. References

Directions

1. Weigh approximately 2 g of sodium benzoate and transfer it to a 25 mL or 50 mL beaker.
2. Record the exact amount of sodium benzoate that you transferred to the beaker. (For example, if your weighing paper and sodium benzoate weighed a combined 3.033 g, and the transfer paper itself weighed 1.050 g, then you used 1.983 g of sodium benzoate.) This information is needed later to determine the % yield of your reaction.
3. Measure 10 mL of deionized water with a graduated cylinder and pour it into the beaker.
4. Use a stirring rod to stir the mixture until the sodium benzoate dissolves.
5. Measure 5.0 mL of 3 M hydrochloric acid (HCl) using a graduated cylinder.
6. While stirring, use a pipet to slowly add the 3 M HCl to your solution.
7. 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.
8. Verify that the pH of the solution is 2 or lower by using the picture on the container as shown below. If it is not, add additional 3 M HCl until you have reached a pH of at least 2. (Be sure to record how much additional HCl was needed.)



9. Cool the solution down to 10 °C or below by setting the beaker in a larger beaker containing ice and water.
10. Separate the solid from the solution using vacuum filtration.
    a. Find a piece of filter paper that fits your Büchner funnel. It should fit flat in the funnel and cover all of the holes.
    b. Slip the filter seal over the stem of your Büchner funnel and then place it on top of the Erlenmeyer flask with the vacuum side arm.
    c. Clamp the Erlenmeyer flask so that it does not tip over during the filtration process.
    d. Attach one end of a piece of thick rubber tubing to the vacuum side arm and the other to the vacuum line. Your setup should look like the one below.



    e. Turn the vacuum line on. Ensure that you have a good seal by placing the palm of your hand (be sure to wear gloves) over the mouth of the Büchner funnel. Check that the filter paper covers all of the holes.
    f. Pour the mixture into the funnel. The solution should drain into the Erlenmeyer flask, while all of the solid should remain in the funnel.
    g. Rinse the crystals with ice-cold solvent (i.e., water).
11. Note the appearance (i.e., color, quality of crystals) of the crude benzoic acid (the solid you just isolated).

12. Recrystallize the crude material to obtain pure product.
    a. Add your crude benzoic acid, 15 mL of deionized water, and a magnetic stir bar to a 50 mL or 125 mL Erlenmeyer flask.
    b. In the fume hood (or under a snorkel), warm the flask and its contents on a hot plate while stirring. At the same time, but in a separate Erlenmeyer flask, heat about 50 mL of deionized water.
    c. Once the both flasks have been heated close to boiling, slowly add hot water to the benzoic acid.
    d. Continue to add hot water to the benzoic acid mixture until all of the solid benzoic acid has dissolved and the solution is clear. (If solid impurities remain in the flask after all of the benzoic acid has dissolved, talk to your instructor about performing a hot gravity filtration.)
    e. Remove the flask from the hot plate, then remove your magnetic stir bar, and allow the flask to slowly cool to room temperature.
    f. If no crystals have formed by the time the flask has cooled to room temperature, then cool the suspension further using an ice bath. Additionally, you can scratch the inside of the flask with a glass stirring rod which often helps to induce crystallization. (If neither of these methods successfully yields crystals, you may have inadvertently added too much water. In this case, boil off a few milliliters of water, and then repeat steps e and f.)
13. Collect the pure crystals via vacuum filtration as described in step 10.
14. Leave the crystals to dry in your drawer until the next lab period.
15. Note the appearance of the product (i.e., color, quality of crystals).
16. Measure the melting point of the dry product.
17. Weigh the dry product.
18. Determine the % yield of your reaction.
19. Record your final conclusions. (What product did you make? What does this imply about the chemical equilibrium of the reaction? Was your initial hypothesis correct? What do you predict happens to sodium benzoate in the acidic environment of the stomach?)

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 20-26.