Preparation of Aspirin

Ap Chemistry Lab

Introduction:

In this experiment, you will be preparing acetylsalicylic acid, or more commonly, aspirin. Doing this experiment will give you a taste of what organic chemistry is all about. The reaction that will take place is illustrated below:

Please note that the points in the structure represent carbon atoms. Many of the hydrogen atoms have been left off to simplify the structures. Also, the circle in the 6-membered ring implies alternating double bonds that show resonance as illustrated below:

This is an example of an ESTERIFICATION reaction. We will come back to this type of reactivity at the end of the year when we get a little more time to talk about organic chemistry.

Procedure:

DAY 1

1. Mass approximately 1.5 g of salicylic acid and place the white powder in a small Erlenmeyer Flask (125mL or smaller). Be sure to record the mass of salicylic acid used in your data table.

2. *Cautiously* add 3 mL of acetic anhydride and then 5 drops of concentrated sulfuric acid.

3. Swirl the reagents and then heat the flask in a beaker of water, warmed to 70-80°C, for 10 min.

4. Remove the Erlenmeyer flask and allow it to cool to room temperature. Add 40 mL of water and cool the mixture in an ice bath to complete the crystallization. The slower and more patient you are with this process, the better your crystals will come out!

5. Filter the crystals away from the solution by pouring the mixture onto a piece of filter paper into a Buchner funnel and aspirator apparatus located at the front of the classroom. The apparatus is pictured to the right. Wash the crystals on the filter with a small amount of ice water. Allow the crystals to drain and then allow the crystals to air-dry.

DAY 2

6. After your aspirin has dried completely, obtain and record its mass.

7. Your product is not likely to be pure (and most certainly is not suitable for human consumption). Among other impurities, most likely, not all the starting material has reacted and is still present in the product mixture. Perform the following test to determine if there is indeed starting material still present. Dissolve a few crystals of salicylic acid and a few crystals of your aspirin in about 5 mL of water (about 1 inch in volume) in two separate small test tubes. Add a drop of 1% ferric chloride solution to each test tube and note the color. Ferric chloride causes a color change in the solution *only* if salicylic acid is present. Record observations in your data section.

8. RECRYSTALLIZATION. This is a process commonly used to separate compounds based on differences in solubility. The next few sentences offer an overview of the recrystallization process. The product mixture is heated and dissolved in water (or some other liquid). The main impurity, salicylic acid, is very soluble in water whereas aspirin is not as soluble. The product mixture is cooled very quickly. The starting material should stay in solution (excess reagents) whereas the aspirin will crystallize out as the temperature lowers. The solid crystals can then be filtered off.

Dissolve the impure acetylsalicylic acid in ethanol (C_2H_5OH) in an Erlenmeyer flask. Use about 3 mL of ethanol for every gram of your impure sample. DO NOT use too much alcohol or your recrystallization will only yield a small amount of product. Heat the Erlenmyer on a hot plate to dissolve the sample.

9. Warm to about 50 degrees Celsius an amount of water that is equal to three times the volume of ethanol you used. Do not overheat the water. Add this water in 5 mL portions to the hot ethanol solution. Swirl the solution to thoroughly mix it between each addition. Cool the solution with a watch glass covering it. When the solution approaches room temp., plunge it in an ice bath.

10. Filter the crystals away from the solution by pouring the mixture onto a piece of filter paper into a Buchner funnel and aspirator apparatus located at the front of the classroom. The apparatus is pictured to the right. Wash the crystals on the filter with a small amount of ice water. Allow the crystals to drain and then allow the crystals to air-dry.

11. You will repeat the purity test and take the final mass next week in lab.

DATA: Make sure to have the following entered into your data table.

1. Mass of salicylic acid used.
2. Mass of *dry* aspirin.
3. color of $FeCl_3$ plus salicylic acid.
4. color of $FeCl_3$ plus crude aspirin
5. Mass of dry purified aspirin
6. color of $FeCl_3$ plus purified aspirin
5. Additional observations. Describe what your crystals look like???

CONCLUSION: Simple, state your results.

ERROR ANALYSIS:

1. Calculate the percent yield of aspirin in your reaction.
   BE SURE TO SHOW ALL CALCULATIONS.

$$\% \text{ Yield} = \frac{\text{Experimental}}{\text{Theoretical}} \times 100.$$

** Moleville calculation...how much aspirin *should* you have made???

2. Explain some errors that might have occur to explain the % yield you obtained.

ANALYSIS & QUESTIONS:

1. In your own words, describe the process of recrystallization and what it accomplishes.
   (a) What major impurity would you be trying to remove with the process of recrystallization??
   (b) Explain how degree of SOLUBILITY is critical to effectively purify the aspirin.
   (c) What role does TEMPERATURE play in this process of recrystallization?

2. List and explain at least TWO reasons why your percent yield of aspirin could be greater than 100%.

3. The reaction your performed was an ESTERIFICATION reaction. Refer to the organic chapter in your text (pg 959 - 961) to answer the following questions:
   a. How can you distinguish an ester. What is it's general format?
   b. How does an ester differ from a carboxylic acid.

4. Research on the internet or in books HOW aspirin works in your body. Phrase your explanation for this process in your OWN words. PLEASE INCLUDE A CITATION FOR YOUR SOURCE!

The End!!! *I sure hope this lab didn't give ya a headache or anything.....get it??? :)*