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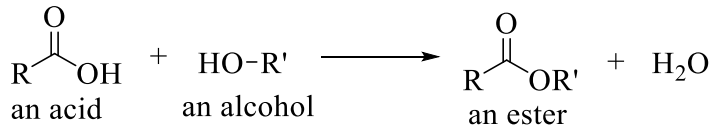
Experiment 14 – Synthesis & Characterization of Acetylsalicylic Acid

Discussion

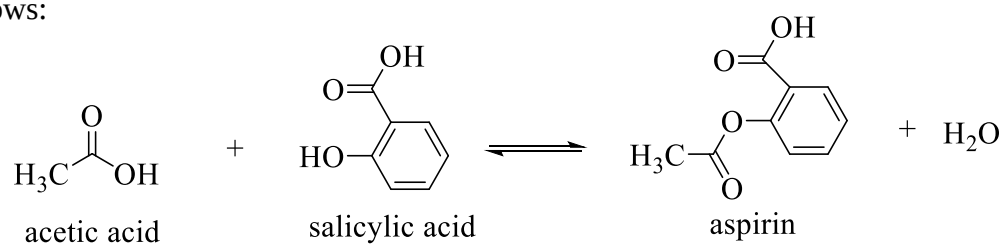
One of the simpler organic reactions that can be carried out is the formation of an ester from an acid and an alcohol. This reaction proceeds as follows:



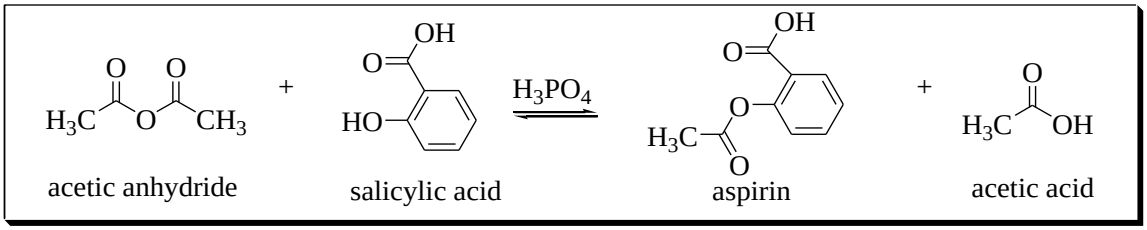
In the equation, R and R' are H atoms or organic fragments like CH_3, C_2H_5, or more complex aromatic groups. There are many known esters in organic chemistry that can be synthesized from organic acids and alcohols. The driving force for the reaction is in general not very great, resulting in an equilibrium mixture of the formed ester, water, acid, and alcohol.

There are some esters which are solids because of their high molecular weight or other properties. Most of these esters are not soluble in water, so they can be separated from the mixture by crystallization. This experiment involves an ester of this type, a substance commonly called aspirin (or acetylsalicylic acid). Aspirin is the active component in headache pills and is one of the most effective, relatively nontoxic, pain killers.

Aspirin can be made by the reaction of the hydroxyl group (–OH group) in the salicylic acid molecule with the carboxylic acid group (–COOH group) in acetic acid. The reaction proceeds as follows:



A better preparative method, which we will use in this experiment, employs acetic anhydride in the reaction instead of acetic acid. The anhydride can be considered to be the product of a reaction in which two acetic acid molecules combine, with the elimination of a molecule of water. The anhydride will react with the water produced in the esterification reaction and will tend to drive the reaction to the right. A catalyst, normally sulfuric or phosphoric acid, is also used to speed up the reaction. The reaction occurs as follows:



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The aspirin you will prepare in this experiment is somewhat impure and should certainly not be taken internally, even if the experiment gives you a bad headache. We will attempt to purify the aspirin via recrystallization with ethanol. If any salicylic acid remains unreacted, its presence can be detected with a 1% iron(III) chloride solution. Salicylic acid has a phenol group in the molecule. The iron(III) chloride gives a violet color with any molecule possessing a phenol group. Notice the aspirin no longer has the phenol group. Thus, a pure sample of aspirin will not give a purple color with 1% iron(III) chloride solution.

Wear gloves and safety goggles! Both phosphoric acid and acetic anhydride are corrosive! They will burn skin! Salicylic acid is also a skin irritant.

Procedure

Prepare a 250 mL beaker of approximately 1/4 full of water. Place it on a hot plate and heat to 80 ºC. Weigh out approximately 500 mg salicylic acid in a 25 mL Erlenmeyer flask. *Perform the next operation in the fume hood*: pipet 1.0 mL of acetic anhydride and pour it into the flask in such a way as to wash any crystals of salicylic acid on the walls down to the bottom. Add 5 drops of 85% phosphoric acid to the mixture to serve as a catalyst.

Clamp the flask in the water bath, and immerse it in the hot water bath for 10 minutes, stirring the liquid in the flask occasionally with a stirring rod. Once the reaction is complete, remove the flask from the water bath, and CAUTIOUSLY add 10 – 20 drops of water to the mixture to destroy any excess acetic anhydride. There will be some hot acetic acid vapor evolved as a result of the decomposition of any unreacted acetic anhydride.

Let the flask cool for a few minutes in air, during which time crystals of aspirin should begin to form. Put the flask in an ice bath to hasten crystallization and increase the yield of product. If crystals are slow to appear, it may be helpful to scratch the inside of the flask with a glass rod. Collect the aspirin by vacuum filtration. Pour distilled water over the crystals; repeat the washing process, and then draw air through the funnel for a few minutes to help dry the crystals. Determine the mass of your impure aspirin.

To purify your synthesized aspirin, transfer it to a 10 mL beaker and add 2 mL of ethyl alcohol using a plastic pipet. Warm the solution to 60 ºC. Cover the solution and allow it to cool undisturbed to room temperature. Then set the beaker in an ice bath and once again scratch the inside of the flask with a glass rod to induce recrystallization. Collect the purified aspirin by vacuum filtration, and let the crystals dry for a few minutes before weighing them. Determine the mass of your dry purified aspirin.

Finally, we will test the purity of your synthesized aspirin with 1% iron(III) chloride solution and compare with a commercial aspirin and salicylic acid. Label three test tubes; place a few crystals of salicylic acid into test tube no. 1, a small sample of your aspirin into test tube no. 2, and a small sample of a crushed commercial aspirin into test tube no. 3. Add 5 mL of DI water to each test tube and swirl to dissolve the crystals. Add 10 drops of 1% aqueous iron(III) chloride to each test tube. Record your results. The formation of a purple color indicates the presence of salicylic acid. The intensity of the color qualitatively tells how much salicylic acid is present.

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Data and Calculations for Experiment 14

Weight of salicylic acid added _____

Volume of acetic anhydride _____

Density of acetic anhydride from CRC _____

Molecular Weight of acetic anhydride _____

Molecular Weight of salicylic acid _____

Theoretical Yield of aspirin _____

Actual Yield of crude aspirin _____

Actual Yield of recrystallized aspirin _____

Percent Yield of recrystallized aspirin _____

Test Tube No.	Sample	Color	Intensity
1	Salicylic acid		
2	Your synthesized aspirin		
3	Commercial aspirin		

Questions

1. Determine the percentage yield of your crude product.

2. 2.0 grams of salicylic acid and 5.0 mL of acetic anhydride (density = 1.08 g/mL) are mixed to produce aspirin. Determine the percentage yield of the reaction if 1.9 g of aspirin is actually obtained in this experiment.

3. In your own words, write a one-half page, well-written abstract of the entire experiment, making sure to briefly state the overall purpose or goal as well as any conclusions. (For additional guidelines on writing this abstract, please refer to the **Moorpark College Chemistry Department Laboratory Report Rubric** found in the lab manual and department website.)