Spectroscopic Aspirin Analysis

Hazard warning: **The sodium hydroxide used in this experiment is highly corrosive. If you get it on your skin, wash immediately. If your skin feels slippery, that is a sign that you have gotten the sodium hydroxide on you.**

Introduction

The objective of this experiment is to determine how much active ingredient, Acetylsalicylic Acid (ASA), is contained in a typical aspirin tablet using visible spectroscopy.

A spectrophotometer is an instrument that can quantitatively measure how much light of a certain wavelength is *absorbed* by a solution. A solution of low concentration will absorb a small amount of light; a solution with a high concentration will absorb more light. This relationship can be exploited to determine how much of a substance is in a solution of unknown concentration via comparison with solutions of known concentration. The absorbance data from the solutions of known concentration are used to produce a "standard curve." A standard curve is a graph of the concentration versus the absorbance at a particular wavelength (see Figure 1). The relationship (i.e., the slope of the line) between these two quantities can then be used to determine the unknown concentration.

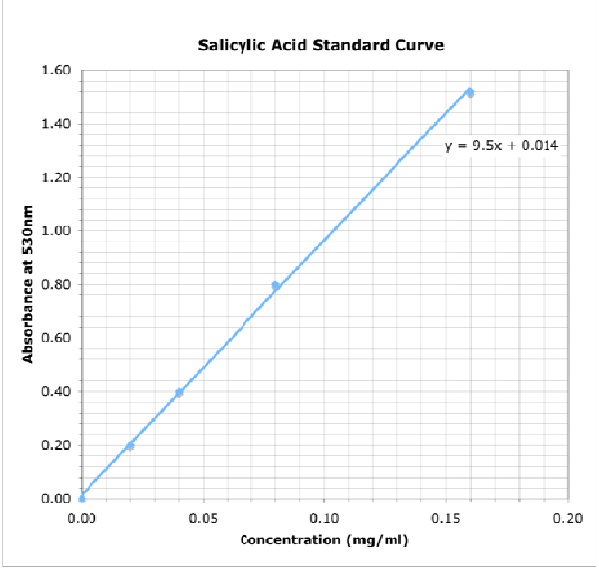

Figure 1

The active ingredient in aspirin is acetylsalicylic acid (ASA- Figure 2). ASA appears colorless in aqueous solution, but a simple chemical reaction will convert ASA into a complex that appears red and absorbs light of 530nm (Figure 3). Since the intensity of the color is directly related to the concentration of the aspirin present, spectrophotometric analysis can be used.

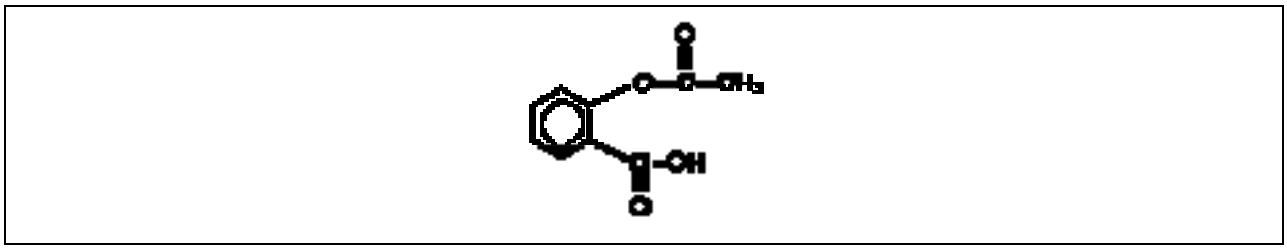

Figure 2- Acetylsalicylic acid (aspirin)

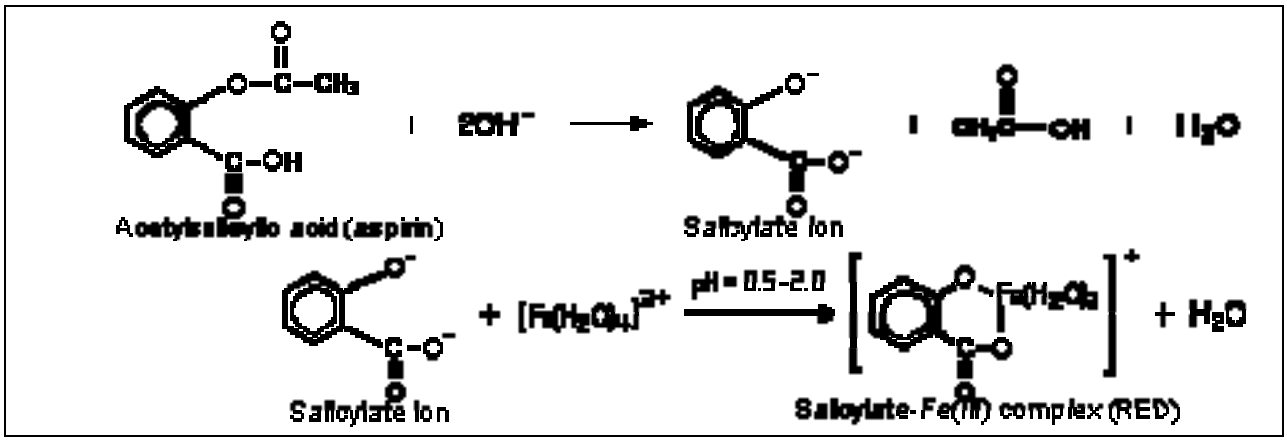

Figure 3- Production of Salicylate-Fe(III) Complex

To produce the colored complex, sodium hydroxide (NaOH) will be used to break the bond between the acetyl group and salicylic acid. This leaves the salicylate^{+2} ion, which will form a complex with Fe^{3+} ions. The salicylate-Fe(III) complex is a dark purple-red color that has a maximum absorption at a wavelength of 530nm (this means the solution absorbs yellow-green light). To determine the amount of salicylate in the aspirin tablet, the absorbance of the solution produced from the aspirin tablet will be compared to the absorbance of "standard" salicylate-Fe(III) solutions (a "standard" solution is one of known composition).

Experiment Procedure

PART 1- Prepare the aspirin samples.

1. Weigh one aspirin tablet to the nearest 0.01g. Record this mass in your lab notebook. Place the tablet in a 125mL Ehrlenmeyer flask.
2. Measure 10mL of 1.0M NaOH solution in a clean, dry graduated cylinder. Add the NaOH to the flask containing the aspirin tablet.
3. Heat the mixture to a mild boil for five minutes on a hot plate to hydrolyze the ASA. Be careful to avoid splattering and do not let the solution dry up to prevent loss of contents. Rinse the inside walls of the flask with a small amount of distilled water to ensure complete chemical reaction of the ASA.
4. Quantitatively transfer the solution to a 250mL volumetric flask through a glass funnel. Thoroughly rinse the flask and funnel with distilled water so that the rinse water flows into the volumetric flask. Add distilled water to the solution in the flask until the bottom of the meniscus touches the index mark of the flask neck. Stopper

the flask. While firmly holding the stopper, invert the flask 10 times to thoroughly mix the solution.

NOTE: The aspirin solution may have a milky appearance due to starch fillers in the aspirin tablet. Some buffering agents like aluminum hydroxide will not dissolve completely in base. If your solution is cloudy, allow the precipitate to settle to the bottom of the flask. When you take a sample of your solution for analysis, use your pipette to remove solution from the top portion of the liquid so that you will not draw any precipitate into your pipette.

5. Label two test tubes "A" and "B". Using a 10ml pipette, measure 9mL of $FeCl_3$ solution and place into tube A. Measure 9.5mL of $FeCl_3$ solution and place into tube B.

6. Switch to a 1mL pipette. Measure 1mL of your aspirin solution and place into tube A. Mix thoroughly by drawing the mixture up into the pipette and squirting it back into the tube several times.

7. Measure 0.5mL of your aspirin solution and place into tube B. Once again, mix thoroughly as described above.

PART 2- Prepare the salicylic acid standard.

1. Label five test tubes 1-5. Take 3ml of $FeCl_3$ solution and place into tube #1. This will be your "solvent blank." It will define what 0% absorbance is in this experiment.

2. Using a 10ml pipette, carefully measure 9ml of $FeCl_3$ solution and place into tube #2. Place 5ml of $FeCl_3$ solution into tube #3; repeat so that tubes #4 and #5 also contain 5ml $FeCl_3$ solution.

3. Using the 1mL pipette, carefully take 1mL of the ASA standard solution and place into tube #2.

4. Switch to a 5mL pipette. In order to thoroughly mix the solution in tube #2 draw the mixture up into the pipette and squirt it back in several times.

5. Take 5mL of the solution in tube #2 and transfer to tube #3; mix thoroughly.

6. Take 5mL of the solution in tube #3 and transfer to tube #4; mix thoroughly.

7. Take 5mL of the solution in tube #4 and transfer to tube #5; mix thoroughly.

PART 3- Spectroscopic analysis of samples.

1. Take your samples and plastic cuvettes over to one of the spectrometers.

2. Following the instructions at the spectrometer, calibrate the instrument using your solvent blank (Tube #1).

3. Measure the absorbance of each sample at 530nm.

4. Analyze your data as described on the report form.