PURDUE UNIVERSITY INSTRUMENT VAN PROJECT

SPECTROPHOTOMETRIC ANALYSIS OF ASPIRIN
(Revised: 1-20-93)

INTRODUCTION

Aspirin is certainly the most widely used medicinal agent ever discovered. Currently 30,000,000 pounds are sold in the United States each year which provides more than 200 five grain tablets for every person in the country. It is effective as an antipyretic drug (i.e. it reduces fever) and as an analgesic, relieving the mild pain of headaches, neuralgia and rheumatism.

The chemical name for aspirin is acetylsalicylic acid. It is an ester derivative of salicylic acid and its common name is derived from the old German version of the name, acetylspirasaeure. Aspirin was first marketed commercially by the Bayer Company in Germany in 1899. The discovery of its important medicinal value was actually a result of earlier studies on the parent compound, salicylic acid, which was shown to have medicinal value, but also some side effects.

The highly colored complex which forms between acetylsalicylic acid (aspirin) and iron(III) chloride enables us to determine colorimetrically the specific amount of acetylsalicylic acid present in a tablet. Since the intensity of the color formed is directly related to the amount of acetylsalicylic acid present, a series of solutions, each with a different concentration of acetylsalicylic acid, can be prepared and the absorbance of each solution measured. A calibration curve is then constructed and from this curve the amount of acetylsalicylic acid in a commercial aspirin product can be determined.

The complex ion is formed in two steps. First the acetylsalicylic acid is reacted with sodium hydroxide to form the salicylate dianion. Then the addition of acidified iron(III) ion produces the violet tetraaquosalicylatroiron (III) complex.

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OBJECTIVES

1.   To use the Spectronic 20 (Spec 20) to determine the milligrams of acetylsalicylic acid (aspirin) in a commercial aspirin product.

2.   To compare the mass and cost of acetylsalicylic acid in various commercial aspirin products.

PROCEDURE

A.   Preparation of Commercial Aspirin Solutions

1.   Weigh out 0.400 g (400 mg) of acetylsalicylic acid and place it in a 125 mL Erlenmeyer flask. Add 10 mL of 1 M NaOH solution and heat to boiling. Quantitatively transfer the solution to a 250 ml volumetric flask and dilute with distilled water to the 250 mL mark. This solution will be referred to as the "Standard Aspirin Solution."

2.   Using a 1 mL graduated pipette, transfer 0.5 mL of standard aspirin solution to a 10 mL volumetric flask or graduated cylinder. Dilute to the 10 mL mark with buffered 0.02 M iron(III) chloride solution. Use a medicine dropper to get the bottom of the meniscus on the 10 mL line. Transfer the solution to a small, clean, dry beaker for mixing and label this "Solution A."

3.   Prepare "Solution B" in a similar manner by transferring 0.4 mL of standard aspirin solution as in step 2 and diluting to 10 mL with buffered 0.02 M iron(III) chloride.

Prepare "Solution C" by diluting 0.3 mL of standard aspirin solution to 10 mL.

Prepare "Solution D" by diluting 0.2 mL of standard aspirin solution to 10 mL.

Prepare "Solution E" by diluting 0.1 mL of standard aspirin solution to 10 mL.

NOTE: If a 1.0 mL graduated pipette is not available, accurately determine the number of drops needed for the delivery of 1.0 mL and then use the appropriate number of drops for the volumes needed for dilution.

4.   Measure the % transmittance of each solution with a Spectronic 20 set at 530 nm. Use a cuvette filled with the pale yellow iron(III) chloride solution for the blank.

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DATA TABLE

Solution	%T	Calculated A *
A		
B		
C		
D		
E		

*The readings for % T are more precise than the readings for the absorbance. Therefore the absorbance should be calculated rather than be read off the instrument.

$$A = -\log T \text{ where } T = \%T \div 100$$

B.   Preparation of Commercial Aspirin Solutions

1.   Place one tablet of the commercial product in a 125 mL Erlenmeyer flask. Add 10 mL of 1 M NaOH solution. Heat and/or stir until the tablet has dissolved. Quantatively transfer the solution to a 250 mL volumetric flask and dilute with distilled water to the 250 mL mark.

2.   As is Step 2 from Part A, use a 1 mL graduated pipette to transfer 0.5 mL of aspirin solution to a 10 mL volumetric flask or graduated cylinder. Dilute to the 10 mL mark with buffered 0.02 M iron(III) chloride solution. Use a medicine dropper to get the bottom of the meniscus on the 10 mL line. Transfer the solution to a small, clean, dry beaker for mixing and label the beaker appropriately.

3.   Measure and record the % transmittance of this solution with a Spec 20 set at 530 nm. Use a cuvette filled with the pale yellow iron (III) chloride solution for the blank.

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DATA TABLE

Commercial Product	%T	Calculated Absorbance*

*The readings for %T are more precise than the readings for the absorbance. Therefore the absorbance should be calculated rather than be read off the instrument.

$$A = -\log T \quad \text{where } T = \%T \div 100$$

CALCULATIONS

Construction of the Calibration Curve (Absorbance vs mg Aspirin/Tablet)

1.   Calculate the concentration (in mg/mL) of acetylsalicylic acid in the "Standard Aspirin Solution."

2.   Using the relationship for dilutions:

        concentration x volume = concentration x volume

        calculate the concentration (in mg/mL) of acetylsalicylic acid for each of the standard Solutions A, B, C, D and E.

3.   Determine the concentration (mg/mL) ratios of the following solutions.

        conc B/conc A   conc C/conc A   conc D/conc A   conc E/conc A

4.   400 mg of acetylsalicylic acid was dissolved and diluted to make Solution A. Since all of the aspirin solutions were dissolved and diluted exactly the same way as Solution A, we can conclude that if the aspirin solution has the same absorbance as Solution A, then the aspirin tablet must have contained 400 mg of acetylsalicylic acid.

        Furthermore, since Solution B has less acetylsalicylic acid than Solution A by the fraction conc B/conc A, we can conclude that this solution could have been made by dissolving:

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$$mg\ of\ acetylsalicylic\ acid\ in\ Solution\ B = \frac{Conc\ B}{Conc\ A}\ x\ 400\ mg\ of\ acetylsalicylic\ acid$$

and diluting it as we did for Solution A. We will call this fraction the "mg of acetylsalicylic acid in Solution B" because, if an aspirin solution has the same absorbance as Solution B we can conclude that the tablet contained this many mg of acetylsalicylic acid.

Calculate the "mg of acetylsalicylic acid" for each of the standard solutions.

5. On a graph with appropriate precision, plot absorbance vs. "mg of acetylsalicylic acid." Absorbance should be on the vertical axis and "mg of acetylsalicylic acid" should be on the vertical axis.

Determination of the mg of Acetylsalicylic Acid Per Tablet

1. Using your data, and the data from other students determine the mg of acetylsalicylic acid per tablet from the calibration curve. Get the other data needed to fill in the table from the appropriate aspirin bottle.

Aspirin Product	Calculated Absorbance	Experimental mg / tablet	Accepted mg / tablet	Cost Per Tablet Tablet

RESULTS AND CONCLUSIONS

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1. **Which tablet provides the highest number of mg of aspirin per dollar? Explain.**

2. **Products such as Anacin® are labeled "extra strength." How many mg of aspirin do these "extra strength" products contain?**

3. **On a cost per mg basis, are "extra strength" products a "good buy?"**

4. **Explain how the Spec 20 was used to relate the absorbance to the concentration of the aspirin.**

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TEACHERS' GUIDE
SPECTROPHOTOMETRIC ANALYSIS OF ASPIRIN

CLASSROOM USAGE

This experiment will work well if students have the necessary quantitative skills. Therefore it is felt that this experiment should be for second semester first year students or second year students. This should not be the introductory experiment for the Spec 20.

This experiment was envisioned as an entire class activity so that many aspirin samples could be analyzed and compared. One or two groups of students would be responsible for making solutions for the calibration curve and other groups of students would be responsible for diluting aspirin samples.

STUDENT TIME

1 class period for solution preparation and 1 class period for data collecting.

MATERIALS

acetylsalicylic acid, reagent grade: each calibration curve needs 0.400 g

> If acetylsalicylic acid is not available, an Anacin® tablet may be used for the standards. The label states that this brand contains 400 mg or 0.400 g of aspirin.

NaOH Solution, 1.0 M: each dilution needs 10 mL

Iron(III) Chloride solution buffered to a pH of 1.6 with HCl-KCl buffer. The calibration curve requires 50 mL; each aspirin dilution requires 10 mL. To make 100 mL of this solution, place 39.5 mL of 0.20 M HCl into 60.5 mL of 0.20 M KCl. Then add 4.15 g of $FeCl_3 \cdot 6H_2O$ (or 3.24 g anhydrous $FeCl_3$).

Reference for buffer: CRC Handbook

Distilled water

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Commercial Aspirin Products

Bayer Aspirin®	Generic Aspirin	Baby Aspirin
Alka-Seltzer®	Anacin®	Bufferin®
Excederin®	Midol®	Extra Strength Aspirin

EQUIPMENT

Spectronic 20 with cuvettes
250 mL volumetric flasks
1.0 mL graduated pipette
10 mL volumetric flasks or graduated cylinders
medicine droppers or dropping pipettes
small beakers (50 mL)
balance and weighing paper

SAFETY AND DISPOSAL Standard Laboratory Safety Procedures

SAMPLE STUDENT DATA

Solution	%T	Calculated A
A	18.2	0.740
B	24.6	0.610
C	35.4	0.452
D	51.2	0.290
E	70.6	0.151

SAMPLE CALCULATIONS

1. Calculate the concentration (in mg/mL) of acetylsalicylic acid in the "standard aspirin solution."

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$$\frac{400 \text{ mg aspirin}}{250 \text{ mL}} = \frac{1.6 \text{ mg}}{\text{mL}}$$

2.   Using the relationship for dilutions:

concentration x volume = concentration x volume

calculate the concentration (in mg/mL) of acetylsalicylic acid for each of the standard Solutions A, B, C, D and E.

Solution A: (0.5 mL)(1.6 mg/mL) = X(10 mL)   X = 0.080 mg/mL

Solution B: (0.4 mL)(1.6 mg/mL) = X(10 mL)   X = 0.064 mg/mL

Solution C: (0.3 mL)(1.6 mg/mL) = X(10 mL)   X = 0.048 mg/mL

Solution D: (0.2 mL)(1.6 mg/mL) = X(10 mL)   X = 0.032 mg/mL

Solution E: (0.1 mL)(1.6 mg/mL) = X(10 mL)   X = 0.016 mg/mL

3.   Determine the concentration (mg/mL) ratios of the following solutions.

conc B/conc A: 0.064/0.080 = .80

conc C/conc A: 0.048/0.080 = .60

conc D/conc A: 0.032/0.080 = .40

conc E/conc A: 0.016/0.080 = .20

NOTE: All solutions are compared to Solution A because all of the aspirin solutions will be diluted as Solution A was diluted. (The aspirin tablet was dissolved and diluted to 250 mL. A 0.5 mL sample of this solution was then diluted to 10.0 mL.)

If you want a more concentrated solution, say 0.6 mL of the stock solution diluted to 10.0 mL, and called this Solution F, the concentration of this solution would be:

Solution F: (0.6 mL)(1.6 mg/mL) = X(10 mL)   X = 0.0960 mg/mL

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and the ratio solution F/solution A = 0.096/0.080 = 1.2

4.   400 mg of acetylsalicylic acid was dissolved and diluted to make Solution A. Since all of the aspirin solutions were dissolved and diluted exactly the same way as Solution A, we can conclude that if the aspirin solution has the same absorbance as Solution A, then the aspirin tablet must have contained 400 mg of acetylsalicylic acid.

Furthermore, since Solution B has less acetylsalicylic acid than Solution A by the fraction conc B/conc A, we can conclude that this solution could have been made by dissolving:

$$mg \ of \ acetylsalicylic \ acid \ in \ Solution \ B = \frac{Conc \ B}{Conc \ A} \ x \ 400 \ mg \ of \ acetylsalicylis \ acid$$

and diluting it as we did for Solution A. We will call this fraction the "mg of acetylsalicylic acid in Solution B" because, if an aspirin solution has the same absorbance as Solution B, we can conclude that the tablet contained this many mg of acetylsalicylic   acid.

Calculate the "mg of acetylsalicylic acid" for each of the standard solutions.

Solution B: 0.80(400 mg) = 320 mg

Solution C: 0.60(400 mg) = 240 mg

Solution D: 0.40(400 mg) = 160 mg

Solution E: 0.20(400 mg) = 80 mg

5.   On a graph with appropriate precision, plot absorbance vs. "mg of acetylsalicylic acid." Absorbance should be on the vertical axis and "mg of acetylsalicylic acid" should be on the vertical axis.

Commercial Product	%T	Calculated Absorbance

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Osco Drug	23.4	.631

Aspirin Product	Calculated Absorbance	Experimental mg/tablet	Accepted mg/tablet	cost per tablet
Alka-Seltzer®	.58	306	324	
Osco Drug	.631	330	325	

ASSESSMENT

1.   Give students an unknown solution and ask them to determine the mg of aspirin that it   represents using the standard curve.

2.   Using the "Standard Aspirin Solution", make a solution which would be equal to 250 mg   of aspirin. Verify this with the data from the Spec 20.

REFERENCE

Central Pennsylvania Association of Chemistry Teachers in Cooperation with NSF, Juniata
     College, and Private Industry. (1990). Student Laboratory Manual for Excellence.