#02 The Aspirin Shelf-Life Scenario

Eugene F. Crabill, East Central High School, St. Leon, IN 47012

INTRODUCTION

The company the students work for produces acetylsalicylic acid, aspirin. They have been assigned the task of determining if different packaging materials and/or environmental storage procedures can increase the shelf-life of the product.

Anticipated Investigative Strategy

The students will be given the background information about aspirin production and the fact that salicylic acid, one of the reactants of aspirin, is considered a production contaminate and is not wanted in the final product. They will also be told that aspirin can and does decompose over time into salicylic acid and acetic acid, thus contaminating the product while on the shelf.

They will also receive information on the colored complex created by the salicylate and Fe^{+3} ions and how to test a colored complex with the Spec 20D. This will give the students the basic information needed for the spectrophotometric testing for salicylic acid.

The students will then set up a monthly testing program using the two types of bottles provided, clear glass/plastic or brown glass/plastic. They will also be asked to consider where aspirin might be stored by the consumer (e.g. medicine cabinet, window sill, refrigerator) and choose one of these three environmental conditions to be tested with bottle type.

Student Audience and Placement in the Curriculum

This lab is designed for my Chemistry II classes. The majority of the students are juniors with a few seniors taking the class also. It will be started at the beginning of the school year so there will be enough time to have 9 monthly testing periods.

Goals for the Experimental Activity

1. The student should become proficient in using the Spec 20D spectrophotometer.
2. The student should become proficient in doing a long-term laboratory exercise.
3. The student should be able to compile experimental data, compare data from different laboratory groups, and analyze the data to formulate experimental conclusions.
4. The student should become proficient in using a computer program to manipulate experimental data.

Developed through the National Science Foundation-funded
Partnership for the Advancement of Chemical Technology (PACT)

STUDENT HANDOUT

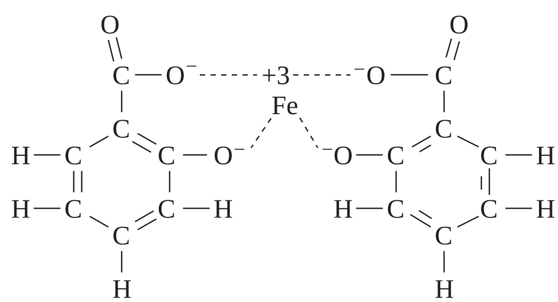
Aspirin Shelf-Life

You have been assigned to a project to determine if the type of bottle aspirin is stored in has any effect on the shelf-life of the aspirin. You will also test for one environmental condition, either temperature or exposure to light, along with the type of bottle. This project will be a long-term testing procedure. You will do an initial test and then monthly testing to determine if the aspirin is breaking down and therefore losing its effectiveness.

Aspirin, acetylsalicylic acid, is produced by the esterification of salicylic acid with acetic acid. Residual salicylate is considered a production contaminate and is not wanted in the final product. The purity of the product depends on the completeness of the reaction and the lack of residual salicylic acid and acetic acid. The aspirin can also decompose over a period of time creating the salicylate and decreasing its effectiveness.

You will test for the salicylate using the Spec 20D. This instrument can determine the concentration of a colored solution by comparing it to an absorption curve for known concentrations of the colored complex. You will prepare the known absorption curve at the correct wavelength and use it for the salicylate concentration determinations.

This colored complex is created by adding Fe^{+3} ions to an aspirin preparation. The free salicylate ions form the complex with the Fe^{+3} ions creating a solution with a violet tint. The intensity of the tint is dependent on the concentration of the complex. The following is a diagram of the complex:



Materials Needed

- standard salicylic acid solution
- (Std. solution is made by dissolving 0.100 g of salicylic acid in 100.0 mL of 50/50 ethanol-water mixture; then diluting 30.0 mL of this solution with enough 50/50 ethanol-water to make 100 mL of solution.)
- ethyl alcohol
- distilled water
- 0.100 M $Fe(NO_3)_3$ solution (stabilized with enough drops of 2.5 M HNO_3 to remove any yellow color)
- commercial aspirin products
- 1 bottle per group; either clear glass/plastic or brown glass/plastic

Safety and Disposal

Goggles and laboratory aprons must be worn during this laboratory exercise.

Ethyl alcohol is a flammable liquid and should not be used near an open flame.

Aspirin can be toxic in high doses. DO NOT taste any chemical or product used in the laboratory. Always wash your hands when you have completed the laboratory exercise.

Ask you instructor for the correct procedure for disposing of the waste material when you have finished the lab.

Standard Absorption Curve Procedure

1. Turn on the SPECTRONIC 20D by turning the power switch clockwise. Allow the unit to warm up for at least 15 minutes to stabilize the source and detector.
2. After the warm-up period, set the wavelength control to 350 nm.
3. Set the display mode to “transmittance” by pressing the mode control until the LED beside “transmittance” is lit.
4. Adjust the display to 0%T with the zero control knob.
5. Fill a clean cell with water and wipe the cell with a tissue to remove liquid droplets, dust, and fingerprints. Place the cell in the sample compartment, align the guide mark on the cell with the guide mark at the front of the sample compartment. Press the cell firmly into the sample compartment and close the lid. Adjust the display to 100%T with the transmittance/absorbance control. Be sure to touch the cell only in the upper 2 cm to avoid putting fingerprints on the cell.
6. Add 10 drops of the 0.100 M $Fe(NO_3)_3$ solution to 50.0 mL of the Std. salicylic acid solution.
7. Press the transmittance/absorbance button until the LED is lit next to “absorbance.” Rinse a clean cell twice with small volumes of the Fe^{+3}-salicylate solution prepared in step #6 and then fill it with the solution to a point approximately 1.5 cm from the top. Follow the steps given in #5 to wipe and place the cell in the unit. Read the absorbance and record on the data sheet for 350 nm.
8. Remove the cell containing the Fe^{+3}-salicylate solution and replace it with the cell containing the distilled water. Adjust the wavelength to 360 nm and the “transmittance” to 100%. Return to the absorbance mode. Put the cell with the Fe^{+3}-salicylate solution in the unit and close the lid. Read and record the absorbance at 360 nm.
9. Continue changing the wavelength by 10nm, resetting the % transmittance to 100%, and read and record the absorbance for the Fe^{+3}-salicylate solution. Your last measurement will be 650 nm.
10. If you are stopping at this point, turn off the Spec 20D by turning the power control counterclockwise until it clicks. If you are continuing to work, do not turn the Spec 20D off.
11. Plot your data (wavelength on the x-axis and absorbance on the y-axis) either on graph paper or on the computer using the graphing program. Draw a best-fit line for each side of the graphed data. The intersection of the lines at the apex will be the wavelength at which the Fe^{+3}-salicylate complex absorbs the most light. This is the wavelength you will use to run the concentration tests. Record this wavelength on the data sheet.

Preparation of Known Concentration Solution

1. If the Spec 20D has been off, follow steps 1-5 in the Standard Absorption Curve Procedure setting the wavelength to the value determined for the Fe^{+3}-salicylate complex. If the Spec 20D has not been turned off, set the wavelength and adjust to 100%T with distilled water.
2. Prepare the following dilutions using your initial Fe^{+3}-salicylate solution as the highest concentration.

   Use a pipet or a self-zeroing buret to measure the Std. salicylate solution and a clean 50-mL volumetric flask for each solution.

Standard Salicylate Solution	Distilled Water	Concentration
5.0 mL	45.0 mL	2.19×10^{-4} M
10.0 mL	40.0 mL	4.38×10^{-4} M
15.0 mL	35.0 mL	6.57×10^{-4} M
20.0 mL	30.0 mL	8.76×10^{-4} M
25.0 mL	25.0 mL	1.10×10^{-3} M
30.0 mL	20.0 mL	1.31×10^{-3} M
35.0 mL	15.0 mL	1.53×10^{-3} M
40.0 mL	10.0 mL	1.75×10^{-3} M
45.0 mL	5.0 mL	1.97×10^{-3} M
50.0 mL (original solution)	0.0 mL	2.19×10^{-3} M

3. Add 10 drops of the $Fe(NO_3)_3$ solution to each dilution and mix.
4. Rinse a clean cell twice and fill with the first dilution. Wipe the cell dry and align it in the Spec 20D.

   Read and record the absorbance on the data sheet. Repeat this procedure for each of the remaining dilutions. Be sure to check the transmittance for 100%T between each dilution with distilled water.

5. Plot the data (concentration on the x-axis and absorbance on the y-axis) on graph paper or on the computer using the graphing program. Draw the best fit line for your data. This will give you the Known Concentration Curve to be used for the rest of the laboratory exercise.

Aspirin Tablet Testing Procedure

1. Crush an aspirin tablet into powder using a mortar and pestle. Place the powder in a 150-mL beaker. Obtain 50.0 mL of 50/50 ethanol-water mixture. Wash the powder remaining in the mortar into the beaker with some of the ethanol-water solution. Pour the remaining ethanol-water solution into the beaker. The salicylic acid and the acetylsalicylic acid will both dissolve in the ethanol-water mixture. The white solid that remains is starch. The starch is used to give bulk to the tablet to insure accurate dosage.

2. Allow the starch to settle out and decant the liquid into another beaker. If the solution is cloudy, filter it using a fluted filter paper. (If you are using "coated" aspirin, the coating will probably be suspended and have to be filtered out. Uncoated aspirin does not seem to have this problem.) Add 10 drops of the $Fe(NO_3)_3$ to the filtered solution and mix.
3. Rinse a clean cell twice and fill with the solution. Follow steps 1-5 from the Standard Absorbance Curve procedure, setting the wavelength to the value determined for the Fe^{+3}-salicylate solution. Read and record the absorbance on the second data sheet.
4. Determine the concentration of the salicylate ion from the known curve. (This will be used as the control concentration.)

5. Obtain a clear glass/plastic or brown glass/plastic bottle. Place one aspirin tablet for each month remaining in the school year into the bottle. Cap the bottle and label it with your initials.
6. Choose an environmental condition:

   refrigerator (cool and dark)
   window sill (warm and light)
   cabinet (warm and dark)
   and place your bottle in that area.
7. Every 30 days, or as close to 30 days as possible, repeat steps 1-3 for a tablet from your bottle. Record your results on the data sheet and make comparisons with the other groups in the class.

8. After the last month's testing, prepare a written report on the results of your tests for salicylic acid present comparing bottle types and environmental conditions. Incorporate the results of the other groups in your class to determine the best bottle type and environmental condition to give the longest shelf-life.

Ideas to Incorporate into Your Report

1. Variance within your aspirin tablets.
2. Variance between groups having the same bottle type and environmental condition.
3. The difference, if any, between bottle types in the same environmental condition.
4. The best bottle type.
5. The best environmental condition for storage.

ASPIRIN SHELF-LIFE

Name______________________________ Date started____________

Class______________________________ Date completed__________

Wavelength Determination			
l	A	l	A
350		510	
360		520	
370		530	
380		540	
390		550	
400		560	
410		570	
420		580	
430		590	
440		600	
450		610	
460		620	
470		630	
480		640	
490		650	
500			

Graphing data shows that the maximum absorbance is at a wavelength of _______nm.

INITIAL SALICYLATE CONCENTRATION

Date____________________ Absorbance__________

Concentration________________

Environmental Storage Condition______________

Monthly Concentration Measurements		
Date	Clear Glass/Plastic	Brown Glass/Plastic

Observations:

INSTRUCTOR NOTES

Aspirin Shelf-Life

Time Required and Group Size

I do this lab with my Chem II classes. I start it at the beginning of September so we can have nine testing periods.

This lab is then used as a second semester project. I have two classes of Chem II each year; the classes range from 14 to 20 students (7 to 10 lab groups). It takes about 45 minutes for 10 lab groups to finish one testing period. The filtering takes the greatest amount of time once they know how to adjust the Spec 20D. Noncoated aspirin works the best if time will be a problem.

Materials Needed

- std. salicylic acid solution (Mixing instructions are on the student handout.)
- ethyl alcohol
- distilled water
- 0.100 M $Fe(NO_3)_3$ solution, stabilized with enough drops of 2.5 M HNO_3 to remove any yellow color
- commercial aspirin products
- 1 bottle per group: either clear glass/plastic or brown glass/plastic

Safety, Handling, and Disposal

The alcohol should not be used near an open flame.

Students should never taste chemicals made or used in the laboratory.

The alcohol/water mixture and the aspirin alcohol/water mixture can be disposed of using the Flinn Catalog disposal procedure #26b. (Be sure your local ordinances allows the disposal of used reagents in this manner.)

Points to Cover in the Pre-Lab Discussion

I go over the use of the Spec 20D and how it works. It is very important that the students understand how to calibrate the Spec 20D and check it during the testing.

We discuss the importance of each group using the same technique when mixing the known concentrations since all groups will be using this data.

We also talk about the importance of “clean” cells with the use of the Spec 20D.

Procedural Tips and Suggestions

You need to draw in the bonds on the two salicylate structures on the first page of the student handout. I am not able to do this with the computer. You can also “box-in” the tables on the student data pages if you would like.

It is important to make sure the students have calibrated the Spec 20D correctly each day it is used. It might be a good idea to have each group have one or two extra tablets in their bottles just in case the Spec 20D is not set up correctly and their data is invalid for a testing period.

I found it very helpful to label the Spec 20D controls and have a copy of the calibration procedure next to the unit. This saves me a good deal of time when they ask how to use it.

You will have to make adjustments to the procedure if your spectrophotometer is not a Spec 20D.

I buy a bottle of 250 aspirin tablets. This is more than enough for the number of groups I have doing the lab.

This is the second year I have done this lab. I found out last year that there is not a great deal of change in the salicylic acid concentration over the school year. The aspirin tablets I bought for the first year,1996-1997, had an exp. date of 02/99 (2.5 years). I have saved last year's data and the aspirin that was not used and we will test those tablets this year and compare the concentrations to last year's results. The tablets I bought this year have an exp. date of 04/99. I will save them for testing next year along with the ones from the first year. I will continue the extended testing as long as there are tablets.

Any good computer program that will handle experimental data will work. I like *f(g) Scholar* and my students are familiar with the program.

For the end of the year summary of this lab we also used TI graphing calculators. It was faster for the students to graph their data and trace the Linear Regression curve to find concentrations for the monthly data. It did take two days to get every one to the point that they could use the calculators. I did have the extra problem of four different models of calculators- TI-82, TI-83, TI-85, and TI-86. It would be easier if every student had the same model.

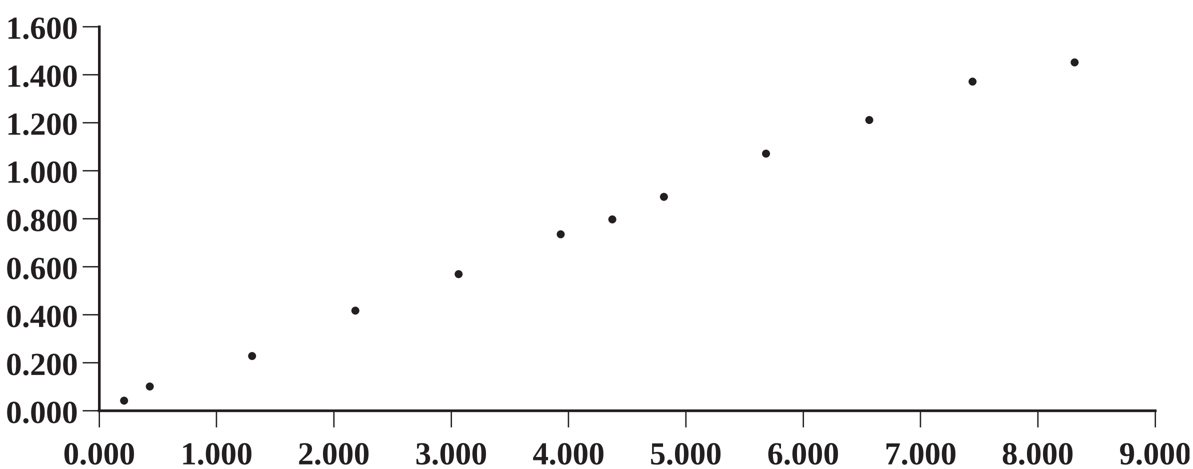
If you have a small group of students doing this lab you may want them to do more than one bottle type and environmental condition. This could be used as a project for Independent Study.

Sample Results

The following is the data my first period class obtained this year for the Std. Absorption Curve. The graph is from the MS WORKS spreadsheet. I could not get the *f(g) scholar* files converted. It does have a much better graph format.

Sample Results	
Molar Concentration ($x10^{-4}$)	Absorption
0.219	0.031
0.438	0.090
1.310	0.217
2.910	0.406
3.070	0.558
3.940	0.724
4.380	0.786
4.820	0.880
5.690	1.060
6.570	1.200
7.450	1.360
8.320	1.440

Std. Curve Raw Data



Examples of the monthly salicylate ion concentrations (x10^{-4}M) collected by the lab groups are as follows:

	September	October	November	December	January	February	March	April	May
Light environment	0.682	0.682	0.828	0.697	0.757	0.779	0.810	0.921	0.896
Dark environment	0.742	0.754	0.670	0.598	0.561	0.706	0.833	0.851	0.978

As you can see there was very little change during the school year. Most groups were disappointed that there was so little change, but understood that this was good in terms of shelf-life.

Extensions and Variations

Some years we make aspirin as part of Organic Chemistry laboratory exercises. I plan on having the students prepare aspirin this year and test their product against the commercial aspirin for purity.

References

Educational Manual Spectronic 20 & 20D. Rochester, NY: Milton Roy Company, 1989, pp 11-14.

Flinn Scientific, Inc., *Flinn Chemical & Biological Catalog Reference Manual 1997,* United States of America, 1997, pp 87,892-893.

Russo, Thomas. "MicroChemistry Teacher's Guide"; West Chester, OH: Kemtec, 1986, pp 102-103.

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