Lesson Plan: Aspirin Synthesis and Spectroscopy Analysis

FOR THE TEACHER

Summary
In this lesson, students will synthesize aspirin and analyze the end product using spectroscopy by applying Beer’s Law.

Grade Level
High School

AP Chemistry Curriculum Framework
This lesson supports the following units, topics and learning objectives:

- **Unit 3: Intermolecular Forces and Properties**
  - **Topic 3.13:** Beer-Lambert Law
    - SAP-8.C: Explain the amount of light absorbed by a solution of molecules or ions in relationship to the concentration, path length, and molar absorptivity.

- **Unit 4: Chemical Reactions**
  - **Topic 4.5:** Stoichiometry
    - SPQ-4.A: Explain changes in the amounts of reactants and products based on the balanced reaction equation for a chemical process.

NGSS Alignment
This lesson will help prepare your students to meet the performance expectations in the following standards:

- **HS-PS1-7:** Use mathematical representation to support the claim that atoms, and therefore mass, are conserved during a chemical reaction.

**Scientific and Engineering Practices:**
- Using Mathematics and Computational Thinking
- Analyzing and Interpreting Data
- Obtaining, Evaluating, and Communicating Information

Objectives
By the end of this lesson, students should be able to

- Determine the amount of product of a reaction that should form based on the stoichiometry of the balanced equation.
- Determine the percentage yield of a reaction.
- Explain the use of visible spectroscopy in determining the concentration of a colored solution.
- Relate Absorbance to concentration based on Beer’s Law.

Chemistry Topics
This lesson supports students’ understanding of

- Chemical reactions
- Stoichiometry
- Visible Spectroscopy
- Beer’s Law

Time
**Teacher Preparation:** 45 – 60 minutes
**Lesson:** 180 minutes
  - Day 1: Synthesis of Aspirin lab activity
Students should read the lab and answer the pre-lab questions prior to arrival. Lab introduction: Use provided PowerPoint presentation (15 minutes). Lab activity (45 - 60 minutes).

**Day 2: Aspirin synthesis wrap up/Spectroscopy introduction**
- Measurement of dried aspirin product/clean up and storage (10 minutes).
- Introductory lesson (20 minutes).
- PhET simulation activity (20 minutes).
- Homework: finish PhET simulation if not completed and read Aspirin analysis lab answering prelab questions.

**Day 3: Aspirin Analysis**
- Lab activity (45 minutes)
- Lab wrap up/Discussion (15 minutes)

**Materials**

- **Synthesis of Aspirin Lab (per group)**
  - Salicylic acid (2.0 g)
  - Acetic anhydride (5.0 mL)
  - 85% Phosphoric acid (5 drops)
  - Watch glass
  - Stirring rod
  - Filter paper
  - Eyedropper
  - Thermometer
  - 400mL Beaker
  - 125mL Erlenmeyer Flask
  - Ice

- **Analysis of Aspirin (per group)**
  - 125 mL Erlenmeyer flask (6)
  - 250 mL volumetric flask
  - 100 mL volumetric flask
  - 600mL of 0.02 M iron (III) buffer
  - Aspirin product
  - 400mg acetylsalicylic acid
  - 20 mL of 1M NaOH
  - 2 Cuvettes
  - 5 mL Graduated Pipet
  - 10 mL Graduated Cylinder
  - Spectrophotometer*

- **Important Materials Notes:**
  - The amounts of materials can be reduced if the acetylsalicylic acid standards are made for the entire class to share. The amount of 0.020 M iron (III) buffer would be 100mL per group and the 1 M NaOH would be 10 mL per group. This would require an additional 500mL of 0.020M iron (III) buffer and 10mL of 1 M NaOH for the class set of standards.
  - *A colorimeter could be used instead of a spectrophotometer.

**Safety**

- Always wear safety goggles when handling chemicals in the lab.
- Students should wash their hands thoroughly before leaving the lab.
- When students complete the lab, instruct them how to clean up their materials and dispose of any chemicals.
Always use caution around open flames. Keep flames away from flammable substances.
Always be aware of an open flame. Do not reach over it, tie back hair, and secure loose clothing.
An operational fire extinguisher should be in the classroom.
When working with acids, if any solution gets on students’ skin, they should immediately alert you and thoroughly flush their skin with water.

Teacher Notes
- This lesson is intended for use in a higher level chemistry class, such as Honors or Advanced Placement Chemistry.
- Prior knowledge in the areas of solution preparation and using a spectrophotometer is not necessary, but may be beneficial for this experiment.
- The lesson is designed to be a series of 2 laboratory experiments that first builds on stoichiometric concept, introducing students to the difference between theoretical and actual yield in an experiment synthesizing a product that the students will recognize. The lesson then takes the experiment one step further after synthesis by analyzing the product created in lab by visible spectroscopy. Students do not often use the product in one experiment in additional experiments. This added layer can relate the idea of quality control in the chemical industry.

Day 1: Synthesis of Aspirin
- Lab Introduction:
  - The Day 1 Aspirin Analysis PowerPoint Presentation (available for download) is an introduction to the lab experiment based on students having the lab and/or lab background to read prior to coming to the laboratory. The students will be able to better discuss and understand the concepts and warnings presented in the PowerPoint. It defines terms such as theoretical yield, actual yield and percent yield. It also provides safety and procedure reminders specific to the synthesis.
  - Prelab questions are designed to be answered prior to coming to the laboratory. Students should have prepared answers as a homework assignment, which can later be changed if incorrect.
  - It is assumed that this is not the first time students would be exposed to the term stoichiometry.
- Lab Activity:
  - The heating source for the experiment can either be a Bunsen burner or a hot plate.
  - Leave the acetic anhydride and phosphoric acid in the fume hood and have students add them to the flask after adding the salicylic acid. Leave 2 designated 10mL graduated cylinders in the hood so fumes are not carried to the lab tables.
  - During crystallization, some students may not have crystals form after being in the ice bath for 5-10 minutes. If this occurs, there are normally 2 reasons. First, the students may have forgotten to add the 20mL of water to the flask prior to placing it in the ice bath. The other reason could be for a lack of surface for the crystals to grow on. In this case add a seed crystal of acetylsalicylic acid to the flask or using a spatula, scratch the bottom of the flask to promote crystal growth.
  - Students can harvest the crystals using scoopulas and microspatulas. This will cause percent yields to be a maximum around 85-90%, but normally around 75%. Students can see why the yield would not be 100%.
  - An optional method to harvest the crystals is to use a Buchner funnel and vacuum filtration. Students will have a higher percent yield, but will still not achieve 100% due to the solubility of the aspirin. Normal filtration is also an option if time permits, but as students continue to add more water, more aspirin will dissolve.
  - Pictures of the reaction apparatus, ice bath and product are below:
o The product must dry overnight and should be checked in the morning for dryness.

Lab Wrap up/Discussion:
- This time is used to help students with calculations, lab questions and preview the day 2 of the lab.

Day 2 & 3: Analysis of Aspirin

- Introductory lesson:
  - The *Day 2 Spectroscopy and Beer’s Law PowerPoint Presentation* (available for download) is an introduction to the lab and concepts (talking points are added to the notes section of the presentation). This PowerPoint has the block diagram of spectroscopy, labeled diagrams of a spectrophotometer and colorimeter, color wheel, Beer’s law and sample graph. Most of the slides are pictures and figures designed so that the individual teacher can use them as talking points to provide the amount of information necessary for the level of the students. Both the spectrophotometer and colorimeter are included so the lab can be individualized for the teacher and the other slide can be deleted. If you have a different spectrophotometer, such as a Vernier Spectrophotometer, it can also be used, but a picture would have to be added to the presentation because the lesson plan creator does not have this instrumentation.
  - **PhET simulation: Beer’s Law:**
    - This simulation is included at the end of the PowerPoint. It is an HTML5 simulation allowing it to be run on Chromebooks as well as PCs. The simulation allows both the light wavelength and test solution to be changed depending on what is selected. This allows students to see how absorbance changes for individual solutions. Since the aspirin/iron complex being analyzed in this lab is a purple solution, teachers should select the KMnO₄ solution to be analyzed. The preset wavelength is 544nm (green) which will reinforce the color wheel that wavelengths opposite colors should be used to analyze the colored solutions. The detector can switch between absorbance and % transmittance, since Beer’s Law is dependent on absorbance, select that choice. The concentration can also be changed. Doing this will show the relationship between concentration and absorbance, and a practice standard curve could be created if the teacher desired. Finally, the wavelength can also be changed reinforcing the idea of wavelength of maximum absorbance.
    - If teachers have more time, or would like to expand on this simulation, further applications/questions can be developed by the teacher to further explore the concept.

- Lab activity:
  - Depending on time, the lab can be completed 2 different ways. The longer version has the students make the standardized aspirin solution from pure acetylsalicylic acid and then perform the dilutions needed for the standard curve. To shorten the time, or to use less materials, the teacher can already have prepared the standardized solutions and dilutions. In either case the students will still need to prepare their aspirin sample. If
students did not prepare an aspirin sample, they could analyze an over the counter aspirin containing product.

- The spectrophotometers must be turned on 15 minutes prior to lab to warm up. Also it is important to instruct the students on proper procedure on using the spectrophotometer or colorimeter that is being used. This could but does not have to include setting the % transmittance, switching that data collection to absorbance, properly zeroing out the blank and then measuring the absorbance of the sample.

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<thead>
<tr>
<th>Material Preparations</th>
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<tbody>
<tr>
<td><strong>Solutions</strong></td>
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<tr>
<td>1 liter of the buffer KCl/HCl buffer solution</td>
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<tr>
<td>0.20 M KCl</td>
</tr>
<tr>
<td>0.20 M HCl</td>
</tr>
<tr>
<td>0.20 M iron (III) buffer</td>
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- Pictures of the lab solutions and standardized solutions are shown below:

- Lab wrap up/discussion:
  - Students may need time to prepare the standardized curve and/or perform the calculations. It is recommended that students use a graphing program that can provide a best fit line and equation of the line so that the concentration can be determined. Both google sheets and plot.ly are adequate to perform this task.

- Homework
  - Depending on the requirements of the individual teacher, two separate or one combined lab report could be generated for this activity.