A two-week project; summary report, and notebook duplicates pages are due at the end of the second week.

**Week I - The Synthesis of Aspirin**

Students work in pairs.

Project 1 will acquaint the student with elementary synthetic techniques and qualitative analysis. In the first lab period aspirin (acetylsalicylic acid) is synthesized from . In the second week, the synthesized aspirin is assayed for percent yield and purity.

**Safety**

Goggles and proper shoes must be worn. The instructor should consult relevant MSDS sheets prior to lab. Dispose of all waste chemicals in the plastic waste container under the hood. Acetic anhydride is a lachrymator, phosphoric acid is caustic.

**Educational Objectives (~60% of grade)**

This lab will be graded based on the results of two lab periods. At that time each student will turn in the following results and others as directed by the instructor. Keep detailed records for the summary report:

1. Synthesize aspirin (ASA) from salicylic acid (SA) and acetic anhydride.
2. Mathematically determine the limiting reagent in the synthetic reaction.
3. Calculate the theoretical yield of acetylsalicylic acid.
4. Calculate the experimental and percent yield of aspirin.
5. Safely handle chemicals and care for laboratory glassware and equipment.

**Lab Notebook Content (20% of lab grade)**

Prior to lab, your notebook should include the title, date, the purpose of the lab and the required data tables. This will provide you with experience in record keeping and proper notebook set up. Your instructor may require that you write an experimental procedure and provide other information.

**Data and observations** are to be recorded in the lab notebook as you perform the experiment. **Calculations** must be workout in detail in the lab notebook at the end of the experiment.

**Pre-Lab Exercise**
CHEM 1211L- Synthesis and Analysis

Complete after you have read the lab introduction and procedures; you may write the answers in your lab notebook.

1. Use your textbook or the CRC Handbook of Chemistry and Physics\(^1\) to find the following information, and then complete the pre-lab question. Record this in your notebook prior to coming to lab.
   - Acetic Anhydride- molar mass and density.
   - Salicylic acid (benzoic acid, 2-hydroxy) - molar mass.
   - Acetyl salicylic acid (benzoic acid, 2-(acetyloxy) (aspirin)) - molar mass.

2. Copy the following data table into your notebook. You will record the data in your notebook during the experiment.

<table>
<thead>
<tr>
<th>What was the initial buret reading?</th>
</tr>
</thead>
<tbody>
<tr>
<td>What was the final buret reading?</td>
</tr>
<tr>
<td>What volume of acetic anhydride was used in the synthesis?</td>
</tr>
<tr>
<td>What mass of salicylic acid is used in the synthesis?</td>
</tr>
<tr>
<td>What is the mass of the empty vial, with the label on and the cap off?</td>
</tr>
<tr>
<td>Mass of the synthesized (wet) aspirin</td>
</tr>
<tr>
<td>What was the limiting reagent?</td>
</tr>
<tr>
<td>What is the theoretical yield in grams of aspirin?</td>
</tr>
</tbody>
</table>

Introduction

\(^1\) The CRC is available in the campus library.
Acetylsalicylic acid (aspirin, ASA) is synthesized from salicylic acid (SA) and acetic anhydride in the presence of an acid catalyst. The Bayer Company of Germany coined the brand name Aspirin in 1899. The history of this drug, its patent rights and trademark is a fascinating story, but too lengthy to discuss here. The name "aspirin" is composed of a- (from the acetyl group) -spir- (from the spiraea flower,) and -in (a frequent ending for drugs during the nineteenth century). Aspirin is a commonly used drug due to its analgesic, antipyretic, anticoagulant, and anti-inflammatory properties.

Aspirin is prepared by the acetylation of the phenol hydroxyl group of salicylic acid.

![Figure 1: Synthesis of Acetylsalicylic acid.](image)

The reaction summary is:

$$C_7H_6O_3 + C_4H_6O_3 \rightarrow C_9H_8O_4 + C_2H_4O_2$$

Procedures
CHEM 1211L- Synthesis and Analysis

Synthesis of Aspirin2 (Time Allotted 60 min.)

1. Prepare a hot water bath using a large beaker filled half way with tap water and heat to boiling. If you are unfamiliar with hotplate settings, try an initial setting of 50% of full scale until your water becomes hot. Then adjust the hotplate setting up or down to maintain a steady boil.

2. Weigh between 1.0 to 1.5x g of salicylic acid on an analytical balance and record the weight to 4-place accuracy (x.xxxx g).

3. From the buret set up under the hood, obtain ~3.xx mL of acetic anhydride in a clean 125 mL Erlenmeyer flask. You must record the initial and final volumes on the buret in your notebook.

4. Add the salicylic acid to the flask and ~3 drops of concentrated phosphoric acid; swirl to dissolve the salicylic acid. Clamp the Erlenmeyer flask to a ring stand and immerse it in the boiling water bath; heat for 15 minutes and stir occasionally with a clean glass rod. While the reaction proceeds, determine the limiting reagent and calculate the theoretical yield of acetylsalicylic acid. These calculations must be completed before you leave lab. Good Lab Practice Always set up the calculations for an experiment in your lab notebook using dimensional analysis before you put any numbers in your calculator.

5. Remove the Erlenmeyer flask from the bath and let it cool to room temperature.

6. Use a glass rod to stir the solution, vigorously rubbing the rod along the bottom of the flask to precipitate the acetylsalicylic acid.

Filtering the Product

1. Set up a Büchner funnel, water aspirator assembly. Place filter paper on the Büchner funnel. Be sure the paper lies flat and covers all the holes. Wet the paper with DI water. Your instructor will demonstrate the set up.

2. Turn on the water aspirator to maximum flow. Pour the solution into the Büchner funnel and collect the aspirin crystals by filtering under suction. Use DI water to remove any acetylsalicylic acid remaining in the flask.

3. Wash the crystals with cold DI water (2-3 mL). Continue the suction for 15 minutes to dry the crystals. Disconnect the rubber tubing from the filter flask, and then turn the water off.

4. Remove the filter paper containing the crystals from the Büchner funnel and place it on a watch glass. Allow it to air dry.

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CHEM 1211L- Synthesis and Analysis

Packaging and Labeling the Product

1. Every product needs a proper label! Be certain to label on the vial with your name, the date, lab period, and the product name and mass.

2. Place the dried sample in a labeled pre-weighed vial. (Record weight with the label on the vial) Determine the “wet mass” of the product. Record this in your notebook.

3. Place the screw cap loose on the top of the vial. The loose cap allows the product to continue drying until the next lab period.

4. Turn in the vials to the instructor.

Clean Up

Wash all glassware and lab equipment that you used with soap and water. Rinse each item with tap water.

**Disposal of Excess Reagents:** Leftover solutions should be discarded in the white carboys under the hood

**Clean up your mess and tidy the lab drawer. Complete the instructor’s data sheet and turn in the duplicate copy of your data table.**

This concludes your work for this lab period. Next lab period you will determine the purity of the aspirin you have synthesized

End of this week’s work.
Students work in pairs

This week’s exercises may be combined with additional lab work. Consult your lab instructor and the lab schedule for detailed directions.

Safety
Goggles and proper shoes must be worn. Chemicals used in this lab may be oxidizing, corrosive, harmful or toxic. The instructor should consult relevant MSDS sheets prior to lab.
Dispose of all chemicals in the plastic waste container under the hood.

Educational Objectives and Expected Results (~70% of grade)
The student will qualitatively determine the purity of the synthesized aspirin and understand how purity affects the yield.

Lab Notebook Content (~20% of lab grade)
Prior to lab, your notebook should include the title, date, the purpose of the lab and the required data tables. This will provide you with experience in record keeping and proper notebook set up. Your instructor may require that you write an experimental procedure and provide other information.

Pre-Lab Exercise
(Complete after you have read the lab introduction and procedures; you may write the answers in your lab notebook. These basic calculations will be on the pre-lab quiz)

1. What is the percent yield of aspirin if the experimental (or actual) yield was 1.050g and the theoretical yield was 2.0516g?

2. If 3.7456g of synthesized aspirin is found to contain a 30.0% impurity of salicylic acid, what is the corrected experimental (or actual) yield?

Introduction
CheM 1211L- Synthesis and Analysis

In this lab exercise, the dry mass of the aspirin synthesized in the previous week and the percent yield will be determined. To determine the purity of the previously synthesized aspirin, a qualitative test will be used to determine if the aspirin has a trace or significant amount of salicylic acid (the starting material) remaining.

The salicylic acid is an impurity in aspirin and produces the effects of nausea sometimes associated with this drug. A chromophore \( \text{Fe}^{3+} \text{ions} \) will attach to the salicylic acid impurity in solution and form a light violet to purple colored solution, due to the \( \text{Fe}^{3+}-\text{salicylic acid complex} \). The intensity of the purple color is proportional the concentration of salicylic acid present in the solution.

Procedures

**Determine the mass of the dry product (allotted time 20 min.)**

Weigh the vial (with top off) containing impure aspirin synthesized in the previous week and record this mass.

**Qualitative Analysis of Aspirin (allotted time 20 min.)**

Materials: Test tubes, test tube racks, labels.

Chemicals: aspirin and 1% Iron (III) chloride (in small bottles)

Analysis of Sample:

Label a clean test tube Sample. Add approximately 5 mL DI water and a very small amount (pin head size) of your aspirin. Follow with 2 drops of 1% \( \text{FeCl}_3 \). Record the color and have your instructor inspect your results and compare it to the Lab Standards. Record the estimated percent of unreacted salicylic acid in the aspirin.

Required Calculations and Clean Up

1. Use the estimated percent of unreacted salicylic acid in the aspirin, to calculate yield of aspirin.
2. Calculate the Percent Yield (by mass) of aspirin.
3. Turn in your vials. Complete the calculations and summary report.
4. Dispose of all solutions in the waste container. Place weigh dished in the trash. Wash test tubes and glassware. You may begin other assigned lab projects.
Summary Report: Synthesis and Analysis of Aspirin (two weeks)

Attach notebook pages with calculations for: The limiting reagent; The theoretical yield in grams of aspirin. The percent yield of aspirin.

<table>
<thead>
<tr>
<th></th>
<th>Possible points</th>
<th>Earned</th>
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</thead>
<tbody>
<tr>
<td>Notebook</td>
<td>15</td>
<td></td>
</tr>
<tr>
<td><strong>Synthesis of Aspirin</strong></td>
<td>40</td>
<td></td>
</tr>
<tr>
<td>What mass of salicylic acid was used in the synthesis?</td>
<td></td>
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<tr>
<td>What volume of acetic anhydride was used in the synthesis?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>What was the limiting reagent for this reaction?</td>
<td></td>
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<tr>
<td>What is the theoretical yield in grams of aspirin?</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Analysis of Aspirin</strong></td>
<td>40</td>
<td></td>
</tr>
<tr>
<td>Analysis of synthesized aspirin: visual appearance of dry product: (instructor’s opinion)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dry mass of the product</td>
<td></td>
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</tr>
<tr>
<td>Estimated percent of unreacted salicylic acid in the aspirin:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Yield of aspirin in grams</td>
<td></td>
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<tr>
<td>Percent yield of aspirin=</td>
<td></td>
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</tr>
<tr>
<td>Yield of aspirin in grams x 100</td>
<td></td>
<td></td>
</tr>
<tr>
<td>theoretical yield in grams of aspirin</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lab Technique (finishes on time, breakage, and sample prep, Proper labeling of sample). Cleaning up area, check out.</td>
<td>30</td>
<td></td>
</tr>
<tr>
<td><strong>TOTAL</strong></td>
<td>100</td>
<td></td>
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</tbody>
</table>
End of Experiment.
Chapter 2 Preparation of a Viscous Solution

Quantitative wet lab; students work individually.

Objectives
The student will become proficient in calculations involving units of concentration, and in the use of the analytic balances and laboratory glassware.

Safety
Goggles must be worn. The materials are nontoxic, but are not to be eaten. Do not leave these materials in a carpet or on furniture.

Lab Notebook Content
Your notebook should include the title, date, purpose, procedure; and data tables. Questions must be workout in detail in the lab notebook. Your instructor must initial this work when you turn in your data sheets.

Prelab Questions
1. What mass is 0.2% of 30 g?
2. If the density of water is 0.996485 g/ml at 27.1 °C, what is its volume of 1.5 g of water at this temperature?

Equipment
Borax
Polyvinyl alcohol
DI Water
Food Coloring
Beakers
Graduated cylinder
Lab balance
Digital thermometer
CHEM 1211L- Synthesis and Analysis

Hot Plate

Introduction

In this lab you will increase your proficiency with the lab glassware and analytical balances by preparing two semi solid solutions.

A solution is a homogenous mixture of two or more components. When substances dissolve in water, an aqueous solution is formed. Water (H\textsubscript{2}O, H-O-H) is a polar molecule because it has an unequal distribution of electronic charge. The electronegative oxygen atom has a greater attraction for electrons than the electropositive hydrogen atoms. The oxygen atom maintains a partial negative charge relative to the partial positive charge on the hydrogen atoms. This uneven charge distribution gives water the ability to dissolve many compounds. The principle of like dissolves like is a useful way to predict if a substance will dissolve in water; polar compounds or those that form pairs of positively and negatively charges ions are likely to dissolve in water to some extent.

Chemicals used for chemistry procedures are called reagents, and must meet standards of purity that specify that contaminates are below a certain level. The assay (often printed on the chemical label), lists the type and amounts of contaminates present. Chemicals that are suitable for use in the laboratory are known as ACS grade (meets American Chemical Society specifications), Analytical Grade or Reagent Grade.

D.I. Water used in this lab to prepare reagents is freed of ions, and particulate matter (dust) by passing tap water through a filter and a bed of mixed resins (the deionizing medium). DI water is preferred for use in laboratory experiments.

Reagent solutions are made by transferring a weighed amount of chemical reagent (solid or liquid) to a volumetric flask, then dissolving it in enough solvent to the calibration mark. Most reagents are dissolved in DI water to form aqueous solutions. The dissolved reagent is called the solute; the DI water is the solvent.

A solution is described in terms of its composition with respect to volume. The composition may be expressed as mass, number of moles or other quantities. The concentration of solution is the ratio of solute per unit volume of solvent. Concentration is a density = mass/volume).
The concentration of a solution can be calculated in several ways:

**Weight to Volume** (w/v), (m/v)

A percent solution that is weight to volume (w/v) or mass to volume (m/v) is prepared by weighing a given mass of solute, and dissolving it in a known volume of solvent. The ratio of the mass of solute to the volume of solvent is the mass percent of the solution, multiplied by 100.

\[
\text{mass} = \frac{\text{Grams of solute} \times 100}{\text{mL of solution}} = \text{mass\% (m/v)}
\]

A solution that contains 10.0 grams of solute in a total volume of 100 mL of solution is a 10% (m/v) solution. Proportional amounts can also be prepared; thus 3.0 g of solute, dissolved to form 30 mL of solution is also a 10% (m/v) solution.

**Weight to Weight** (w/w), (m/m)

This type of percent solution designates the mass of solute per 100 g of final solution. If 10.0 grams of solute are dissolved in 90 g of DI water the resulting solution is 10% (m/m).

Considerable ambiguity can arise when a label designates a percentage concentration, but fails to specify if weight to volume (w/v) or weight to weight (w/w) units of concentration apply. An example is a commercially prepared, aqueous solution of hydrogen peroxide (used for cleaning wounds and bleaching) which gives the concentration of hydrogen peroxide as “3%”.

**Molarity** (moles/liter)

The SI unit for expressing the number of molecules in a solution that are available to react with other molecules is moles of solute per liter of solution and is called the **molarity** of the solution. This concentration unit is expressed as mol/Liters or mol/1000 mL of solution.

The mole is the amount of a substance that contains Avogadro’s number of objects $6.022 \times 10^{23}$, thus a one molar solution (1 M) contains Avogadro’s number per liter. A 0.6 M solution will contain 60% of Avogadro’s number per liter of solution or $1.813 \times 10^{23}$ objects per liter.

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3 Percent = per 100.
When a solution is prepared by diluting a stock solution of known concentration, there is a mathematical relationship between the concentration of the stock solution and the diluted solution. This relationship is given by the dilution equation:

\[ M_{stock} \times V_{stock} = M_{dilution} \times V_{dilution} \]

Where:
- \( M_{stock} \) is the concentration of the stock solution
- \( V_{stock} \) is the volume of the stock solution
- \( M_{dilution} \) is the concentration of the diluted solution
- \( V_{dilution} \) is the volume of the diluted solution

**Example 1:**

What mass of sodium chloride is needed to prepare 90.0 mL of a 0.35% (w/v) solution?

This problem is solved by recognizing that 100 mL of a 0.35% solution contains 0.35 g of solute. A solution of 90 mL must contain a proportional mass of solute. Thus:

\[ \frac{0.35 \text{ g}}{100.0 \text{ mL}} = \frac{X \text{ g}}{90.0 \text{ mL}} \]

Solving for \( X \) we find that the required mass.

\[ 0.35 \text{ g} \times 90.0 \text{ mL} = X \text{ g} = 0.32 \text{ g} \]

\[ \frac{0.35 \text{ g}}{100.0 \text{ mL}} \]
Example 2:
What mass of sodium chloride is needed to prepare 100.0 g of a 0.35% (w/w) solution?

The total mass of the solution is to be 100g. We will assume that the density of the water is 1g/mL.

\[
100\text{g} - 0.35\text{g} = 99.65\text{g}
\]

Dissolve 0.35 g of sodium chloride in 99.65 g (~99.65 mL) of water to make this solution.

Preparation of Slime (mass/volume percent)

*Slime*, a traditional Halloween party favor, is a *polymer of polyvinyl alcohol*. It is used to create eerie ambience and for frightening parlor tricks.

In a beaker prepare 25 g of 4% (w/w) sodium borate solution.

Calculate the mass of sodium borate required to prepare the solution.

Heat the solution and stir with a clean glass rod until all sodium borate is completely dissolved. Rinse and dry the stirring rod.

In a beaker, prepare 50 mL of a 10% (w/w) polyvinyl alcohol (PVA) solution.

Heat the DI water to ~80°C. Gradually transfer the PVA to the hot water with stirring until the solid is dissolved. Add a few drops of food coloring, and allow the solution to cool to ~30°C.

Add the sodium borate solution to the PVA solution and stir vigorously until a viscous gel clings to the stirring rod. The glue molecules join together to form large molecules called polymers. The sodium borate acts as the cross linking agent or "connector" for the polyvinyl alcohol molecules causing them to stick together.

Describe the both the appearance and consistency of the solution result from a mix of 10% (m/v) polyvinyl alcohol and 4% sodium borate.
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Package the gel in a Ziploc® bag. You may take it home; it is nontoxic. Store it in the refrigerator.

At home, Elmer’s glue (a solution of PVA) and commercial borax make an excellent slime. See the internet for recipes.

Critical Thinking
1. What changes would you make to produce slime with a drier, more rubber like consistency? Experiment on your own by changing the concentration of one of the reagents. Make additional solutions of the sodium borate solution or the PVA solution at a concentration that you choose. Describe your results in your notebook.

Clean Up
Wash and dry all glassware including the vials. Return the vials to the cart. Turn off thermometers and return them to the instructor. Unwanted slime goes in the trash can.

Unwanted slime- to the trash can.

End of Experiment