Today we’re going to synthesize a useful substance: aspirin. Lewis structures help us understand the molecules that define that substance. They help us understand how the molecule behaves and how the reaction (chemical change) occurs.

Your goal is to conduct this synthesis and then analyze the products with IR and melting point techniques.

You will report the yield and purity of your final product.
Exp 14: Synthesis of Aspirin

Acetic Anhydride

Acid Catalyst

Salicylic Acid

Acetic Acid

Acetylsalicylic Acid
Procedure:
At the beginning of class heat a 400 mL beaker of water on a hot plate to 70-80 degrees at your lab bench top station.
1. Weigh a clean, dry, 125 mL Erlenmeyer flask with a rubber stopper and record its mass.
2. Add approximately 2.0 g of salicylic acid to the flask and weigh it again. Record the mass to the nearest 0.01 g.
3. In the fume hood, add approximately 3.5 - 4.0 mL of acetic anhydride to the flask containing the salicylic acid and stopper it. Weigh it again. Record the mass to the nearest 0.01 g.
4. In the fume hood, add five drops of concentrated acid to the flask containing the salicylic acid and acetic anhydride and swirl to mix everything thoroughly. Either concentrated sulfuric or phosphoric acid may be used.
5. Clamp the stoppered Erlenmeyer flask to a ring stand to hold it in the 70-80˚ C water bath. Heat it for fifteen minutes with occasional stirring. If solid remains, heat it for an additional fifteen minutes.
6. Remove the flask from the water bath and slowly add ten drops of deionized water to convert any excess acetic anhydride to acetic acid.

7. Add about 10 mL of ice-cold deionized water to the flask and cool in an ice bath until crystallization appears to be complete. If crystals do not appear scratch the walls of the flask with a stirring rod to induce crystallization.

8. Assemble a Büchner funnel and filter flask as shown in the figure.

9. Filter the crystals by vacuum filtration. Rinse the residue on the filter paper with 2 mL of ice-cold deionized water. Be aware that using too much water or warm water in this step may affect your results. Dispose of the filtrate in the appropriate waste container.

10. Remove a pea-size amount of the crystals from the Büchner funnel and set aside to determine the melting point and infrared spectrum of this crude sample.
11. Purify the rest of the aspirin by recrystallization. Using a clean, dry 150 mL beaker, dissolve the aspirin in about 1mL of 95% ethyl alcohol, and warm the mixture slightly by placing it in the hot water bath.

12. Stir to dissolve it completely, and then add 5 mL of warm (70°C) deionized water.

13. Cool the mixture in an ice bath until recrystallization is complete.

14. Weigh a piece of filter paper and vacuum filter the product. Allow it to dry on the filter paper until the next lab period. Dispose the filtrate in the appropriate waste container.

15. When your aspirin is dry, record the mass of the filter paper + aspirin. Determine the mass of the aspirin.

16. Determine the melting point of your aspirin sample (Your instructor will demonstrate how to use the melt-temp apparatus.)

17. Generate an infrared spectrum of your aspirin sample (Your instructor will demonstrate how to use the FT-IR.)
Exp 14: Synthesis of Aspirin

Melt-Temp Settings:
- START: 60 °C
- RAMP: 5
- STOP: 150 °C

The melting point of aspirin is 136°C.
Exp 14: Synthesis of Aspirin

Salicylic Acid (SA)

\[
\text{PhCOOH} + \text{CH}_3\text{COOCH}_3 \rightarrow \text{PhCOOCH}_3 + \text{CH}_3\text{COOH}
\]

Salicylic Acid
Exp 14: Synthesis of Aspirin

Acetylsalicylic Acid (ASA) (Aspirin)

\[
\begin{align*}
\text{Acetylsalicylic Acid} & \rightarrow \text{Products} \\
\text{Acetylsalicylic Acid} & + \text{Compounds} \rightarrow \text{Products}
\end{align*}
\]
Exp 14: Synthesis of Aspirin

50/50 Mixture of SA & ASA

\[ \text{Salicylic Acid} + \text{Acetic Anhydride} \rightarrow \text{Acetylsalicylic Acid} \]
Finding the signature peaks of Aspirin.

Ester carbonyl.

Benzene double bond.

Acid carbonyl (conjugated).

\[
\text{Acetylsalicylic Acid}
\]
Questions?