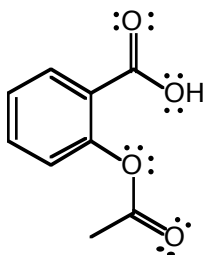


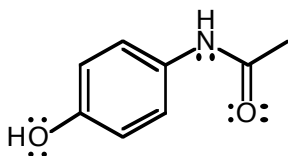
Analysis of Pain-relieving Medications by TLC

Introduction

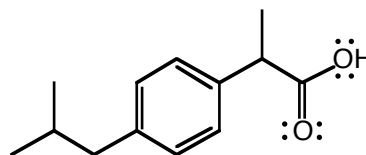
In this lab you will learn to use TLC to identify the biologically active compounds in several over-the-counter pain relievers. The three most common OTC pain-relieving compounds are aspirin, acetaminophen (found in Tylenol), and ibuprofen (found in Advil). Their structures are shown below.



aspirin



acetaminophen



ibuprofen

We will prepare solutions of these compounds and compare them by TLC, then analyze solutions of Anacin, Excedrin, and Smith's Headache Medicine to see which compounds each of these products contain.

Each group will prepare one of the solutions and everyone in that section will use them. You will then test the three standards (aspirin, acetaminophen, and ibuprofen) using three different developing solvents – ethyl acetate, hexanes, and a mixture of these that you will choose. Finally, you will test each of the three products against the three standards to see which they contain.

To prepare for this lab, please read all of the instructions for this lab, as well as "Analyzing a Mixture by Thin Layer Chromatography (TLC)." Write an introduction in your lab notebook. Then answer the pre-lab questions on-line.

Procedure

Preparation of the solutions to be analyzed:

- Select one of the products to be tested and crush one tablet using a mortar and pestle. Discard any outer coating (especially if it is colored), then pour the crushed powder of the tablet into the labeled 50 ml flask on my desk.
- Add 15 ml of ethanol and 15 ml of dichloromethane (use 10 ml of each for ibuprofen and 20 ml of each for aspirin) and stir vigorously. This will dissolve the active ingredients (which are soluble in organic solution) out of the binders (which are water soluble but not soluble in organic solution). Let the insoluble material sink to the bottom.

Analysis of the standard solutions:

- Prepare three TLC plates by drawing a line across the bottom with a pencil, then adding 4 hash marks each, one for aspirin, one for acetaminophen, one for ibuprofen, and one for a mixture of the three.

- Put the first solvent, ethyl acetate, in your 50 ml beaker and check to make sure you have drawn the line properly. Cover the beaker with a watch glass to minimize evaporation of the solvent.
- Spot the first three plates with each of the solutions according to your marks, cleaning the capillary tube between each one. As you do so, check the plate under UV to make sure your spots are visible but not too big (they shouldn't overlap). If they aren't bright enough, spot them again, making sure to let the solvent evaporate before spotting.
- Develop the first plate in ethyl acetate, removing it when the solvent is near the top.
- Visualize it by UV light, tracing the spots that you see with a pencil. Copy it onto your notebook, making it the same size as the plate itself. Make note of any interpretations of this data. Which compound is the most polar?
- Change the developing solvent to hexanes and develop another plate and visualize it. Compare the two solvents. Which was more polar? Copy it into your notebook.
- Using the results of the two previous plates, decide on a mixture of ethyl acetate and hexanes that you think will carry the spots to the middle of the plate. Prepare this mixture, and develop a third plate in the mixture that you have chosen. Copy this plate into your notebook. Once the plates have been copied, you can throw them away.

Analysis of the commercial products:

- Prepare 3 TLC plates, one for each commercial product - each one should have a spot of the solution you are analyzing and a spot for each of the three standards.
- Develop each plate in the solvent mixture that you have chosen and visualize it by UV. Determine what active ingredients each of the products contains.
- Copy these plates into your notebook. After you have finished your analysis, look at the ingredients listed on the bottles and see if they match your analysis.
- Write a conclusion in which you discuss the results of your unknown TLC's, comparing them to the ingredients listed on the bottles. Make note of any additional spots.

Questions

- 1) What does it mean to develop a TLC plate? What does it mean to visualize the plate?
- 2) What will happen to your TLC plate if the solvent in the beaker goes above the line where the solutions are spotted?
- 3) If the developing solvent evaporates off of the plate as the TLC is running, the spots will all run together, and the resulting TLC will be very difficult to interpret. What should you do to prevent this?

- 4) If you ran a plate containing spots of aspirin, acetaminophen, and ibuprofen in ethanol, would you expect the spots to run near the top or near the baseline? How about if you ran it in toluene? (Use your chart from the Physical Properties lab.)
- 5) Spots that make long streaks up the plate are probably the result of solutions that are too concentrated. If this happens, what can you do to alleviate the problem?
- 6) Three of the commercial products contain an additional ingredient which should appear just above the baseline. Look at the labels and determine what it was. Was it more or less polar than the other compounds? Look up its structure in the Aldrich catalogue and draw it.
- 7) The three compounds that we analyzed in this experiment all showed up under UV light. Not all organic compounds do this. What do the structures of these three compounds have in common that would cause them to absorb UV light?