

Date: March 2, 2018

Objectives:

The objective of this experiment was to use Thin-Layer Chromatography to analyze and separate common Analgesics. Using TLC plates coated with silica gel, known and unknown solutions we can analyze the components of Analgesics by using a developing chamber, solvent, and UV light and determining R_f values. R_f value is a measure of how far a component of the mixture moved relative to the solvent front. We also used Bunsen burners to heat and draw out four micropipettes from open ended capillaries.

Data/Observations:

Two TLC plates were prepared. One with the following standards: mixture of aspirin, acetaminophen, and caffeine, the second with acetaminophen, and the third with aspirin. We determined the caffeine R_f by comparing both the aspirin and acetaminophen R_f values. The second TLC plate contained Anacin and Kroeger PM which was our test for the unknown components of each product.

A: Analysis of Analgesics:

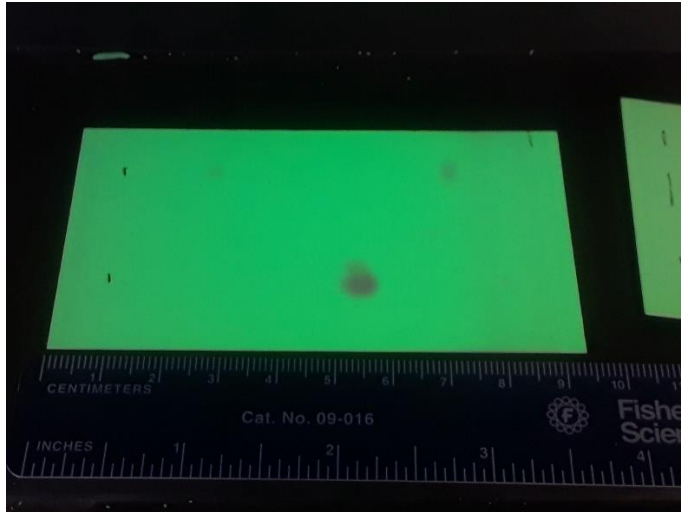
After preparing four micropipettes, a water bath was prepared. Anacin and Kroeger PM were each crushed using a mortar and pestle. 5 mL of 95% ethanol was added to the mortar and then the mixture was placed into a large test tube. This was done for each analgesic separately. The test tubes were then allowed to simmer in a water bath. Heat was used to separate the organic components from the inactive ingredients. The particulate at the bottom of each tube was the inactive.

Two TLC plates were prepared. The first TLC plate had Anacin and Kroeger PM. We had to do this a couple of times to adequately pipette enough of each product to be visualized under the UV light. The second TLC plate was prepared with the mixture of three compounds, acetaminophen, and aspirin.

A developing chamber was set up with a 500 mL beaker, a watch glass and paper towel. 10 mL of the developing solvent were placed into the 500 mL beaker.

UV light was used to view and measure both TLC plates. Measurements and findings were recorded.

R_f Value 0.00 – 1.00. R_f = distance a / distance b. I used 8.5 cm as the distance from initial spot to solvent front. 1/8.5 = .12 per centimeter. I multiplied measurements by .12.



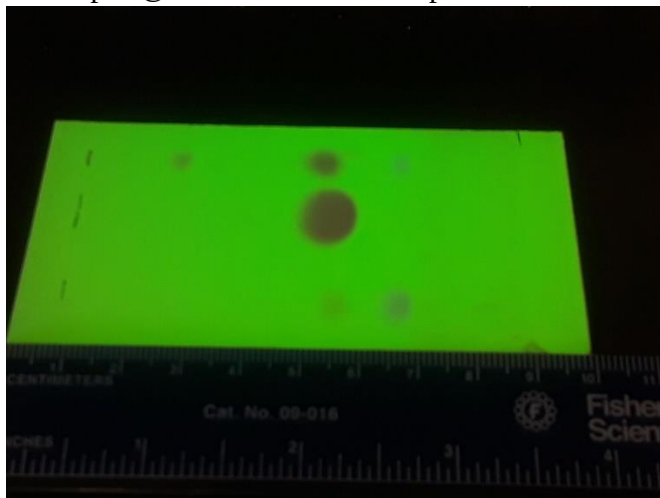
Top = Anacin

First spot @ 2.5 cm = caffeine – R_f .30

Second spot @ 6.75 cm = aspirin – R_f .81

Bottom = Kroeger PM

First Spot @ 5.5 cm = acetaminophen – R_f .66



Top = Mixture

First spot = caffeine – 2.5 cm = .30 R_f

Second spot = acetaminophen – 5.5 cm = .66 R_f

Third spot = aspirin – 6.75 cm = .81 R_f

Middle – Acetaminophen =

Bottom - Aspirin

Conclusions & Study Questions

Conclusion:

After finishing the experiment, I was curious as to why the Kroeger PM only showed signs of Acetaminophen, believing Kroeger PM was a generic for Excedrin PM, which contains both aspirin and acetaminophen. A quick check online proved that Kroeger PM contained Acetaminophen and Diphenhydramine, while Excedrin PM contained all three: Acetaminophen, aspirin and Diphenhydramine. We have two spots on our standards for aspirin. We had five compounds that we pipetted and four pipettes. I believe the extra spot below aspirin was from using the same pipette twice and captured some acetaminophen left in the pipette. It also matches the order in which we put the compounds onto the TLC plates.

Study Questions:

- 1. Why are both polar and nonpolar compounds eluted by polar solvents, but only nonpolar compounds are eluted by nonpolar solvents?**

Because nonpolar compounds are not adsorbed as strongly. Polar compounds would stick to the silica gel, which is very polar. A nonpolar compound wouldn't be able to carry it.

A nonpolar solvent wouldn't bind to the adsorption sites, which are polar. The polar compound would bind to the sites since they would be available. The nonpolar solvent wouldn't be able to carry the polar compound but would carry the nonpolar compound.

- 2. What will be the result of each of the following errors in TLC technique?**

- Solvent reservoir in developing chamber too deep.
This would interfere with adsorption and carrying, and possibly dilute or distort the compound.
- Solvent front allowed to rise only half of the way up the TLC plate.
The solute won't be able to complete the process. It will stop too soon and the R_f value will be inaccurate.
- Solvent Polarity too low.
If the polarity is too low it won't carry or elute well.
- Solvent polarity too high.
Everything will move up to the top of the plate very quickly and things might not separate out properly.
- Faint spots applied at the origin.
Compound leaked or didn't stay in one place.

3. **Identify the active ingredients in your unknown analgesic tablets. Explain whether the ingredients are appropriate for the intended purposes of the particular preparations.**

Anacin – contained aspirin and caffeine. Kroeger PM, acetaminophen.

Ingredients are standard. Not all aspirin contains caffeine, but a lot of brands do add it.

4. **Imagine an experiment in which a mixture composed of the following five compounds is subjected to TLC analysis.**

Ph-OCH₃, Ph-COOH, Ph-CH₂CH₃, Ph-CH₂OH, PhCOCH₃.

Ph = C₆H₅. Benzene ring with O.

Increasing R_f. Polar to Nonpolar.

- Ph-COOH – Carboxylic Acid - Polar
- Ph-CH₂OH – Alcohol - Polar
- Ph-COCH₃ – Ketone – Somewhat Polar
- Ph-OCH₃ – Ether – Not very Polar
- Ph-CH₂CH₃ – Alkane – Not very Polar – Highest R_f.

5. **Consider the TLC experiment in (4) above. What, if any, differences would be observed if ethanol were used as the eluent in place of benzene?**

Ethanol is more polar than benzene, which is only slightly polar. Compounds will move farther the more polar the solution. The higher the polarity of a solvent, the more effectively it will carry/elute both polar and nonpolar compounds. I would expect a higher R_f value if ethanol were used as the eluent.