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Experiment 15: Thin-Layer Chromatography Analysis of Drug
Components

March 4, 2019

Purpose:

The purpose of this experiment is to use TLC to determine components of the analgesic drug that is assigned.

Equations: NONE

Mechanisms: NONE

Amounts and Properties:

Table 1: Chemicals and important properties

Chemical	Amount
Ethyl Acetate/Acetic Acid	5 mm equivalent in chamber
Ethanol/DCM	2.5 mL

Hazards and Safety:

DCM is harmful if ingested, inhaled, or absorbed through skin. May cause cancer if prolonged inhalation. Minimize contact with ethanol/DCM solvent mixture and standard solutions and do not breathe vapors. Ethyl acetate is flammable and may be harmful if inhaled or absorbed through skin. Avoid contact and don't breathe vapors. Wear gloves and handle everything under the hoods. To dispose: Dispose all in labeled containers under the hood.

Procedure:

Prep Of Chamber:

1. Use ethyl acetate/acetic acid (200:1) as developing solvent.
2. Filling developing chamber with paper wick and about 5 mm of solvent.
3. Cover with a lid and slosh up sides to moisten wick, and put under hood.

Prep and Development of TLC Plate:

1. Take unknown and crush a quarter into a powder and remove any coating it had.
2. Transfer power to test tube and add 2.5 mL of 1:1 ethanol/DCM.
3. Mix to dissolve as much as possible.
4. Use pipet to take solution to a small vial leaving behind any solid and cap the vial.
5. Spot the unknown and all standard solution on a silica gel TLC plate with fluorescent indicator and label spots. Avoid cross contamination.
6. If possible spot unknown in different concentrations at 2 or more locations.
7. Develop under hoop in chamber and don't disturb it. Mark solvent front before plate dies up.

Visualization and Analysis:

1. When plate is dry observe spots under 254 nm wavelength UV, outline with pencil and mark most intense spot for each.
2. Visualize under Iodine vapor or another reagent if needed.

Observations:

Each plate took about 15 minutes to develop and for the unknown that was acquired, the spots were about $\frac{1}{3}$ of the way up the plate. The aspirin plate went up further along the plate while the acetaminophen was about the same with the unknown sample.

Measurements:

Table 2: Distances for first plate with aspirin

Location of Spot	Distance from solvent front (9.5 cm long)
Lane 1	3.5 cm
Lane 2, First Spot	3.5 cm
Lane 2, Second Spot	6.5 cm
Lane 3	6.5 cm

Table 3: Distances for second plate with acetaminophen

Location of Spot	Distance from solvent front (9.5 cm long)
Lane 1	3.6 cm
Lane 2, First Spot	3.8 cm
Lane 3	3.8 cm

Data and Calculations:

$R_f = \text{distance by component} / \text{distance by solvent}$

 R_f for Table 2:

$$\text{Lane 1: } \frac{3.5 \text{ cm}}{9.5 \text{ cm}} = .37$$

$$\text{Lane 2, Spot 1: } \frac{3.5 \text{ cm}}{9.5 \text{ cm}} = .37$$

$$\text{Lane 2, Spot 2: } \frac{6.5 \text{ cm}}{9.5 \text{ cm}} = .68$$

$$\text{Lane 3: } \frac{6.5 \text{ cm}}{9.5 \text{ cm}} = .68$$

Rf for Table 3:

$$\text{Lane 1: } \frac{3.6 \text{ cm}}{9.5 \text{ cm}} = .38$$

$$\text{Lane 2: } \frac{3.8 \text{ cm}}{9.5 \text{ cm}} = .4$$

$$\text{Lane 3: } \frac{3.8 \text{ cm}}{9.5 \text{ cm}} = .4$$

Discussion:

After conducting the experiment, the Rf values that were acquired from the first plate with aspirin were .37, .37, .68, and .68 respectively. This shows that there were no aspirin contained within this sample. For the second plate, the values were .38, .4, and .4 respectively. This demonstrates that there is acetaminophen within the samples that were acquired.

Conclusions:

Since there is acetaminophen within the sample from the conclusion of the TLC plates, there were also no other spots seen on the TLC plates. Since Excedrin has both aspirin and acetaminophen, this would not be the correct answer being that the sample did not show an aspirin spot during the plate analysis. The only other choice would be Tylenol which only has Acetaminophen and that matches the plate analysis. Any sources of error could be that when spotting, the spots were too big that could've contaminated the other lanes, having the plate touch the side of filter paper within the chamber, or even just mixing up which lane had what substance.

Exercises:

1. Phenacetin would have a higher Rf value because of the structure being more nonpolar than that of acetaminophen. Since the developing solvent is nonpolar, the molecule that is closer to being nonpolar would have a Rf value due to the "like dissolves like" phrase.
2. The solid that would be left behind is the coating on the crushed pill. The pill coating is not an active ingredient thus not dissolving with the solvent.
3. A) There could be two possibilities, one would that the Rf value that is calculated would be incorrect, or that the samples could move higher than it should have. B) Since Ammonia is basic, the hydroxyl group can react with the ammonia and not show any sign of movement on the chromatogram. C) The spots would be too big if an open-ended melting-point capillary was used. These spots would be able to dilute each other causing the experiment to have wrong data. D) Using a pen would have the ink migrate up as well causing the experiment to yield wrong data.