30 Aug 2024

Separation of Analgesic Drugs using TLC

Purpose: Thin Layer Chromatography can be used to help distinguish different components in a solution. This Exp. will be used to determine if a "mystery powder" is Advil, Aspirin, Tylenol, Anacin, or Excedrin.

Needed supplies:

Materials:
- Lead pencil
- PPE (Gloves, glasses, lab coat)
- 400 mL Beaker
- 2 TLC plates
- watch glass
- 13 x100 Test Tubes
- Filter
- UV lamp
- Iodine Chamber
- Ruler

Chemicals
- 2 mL Ethyl acetate *
- 1.8 mL Glacial acetic acid *
- 1.8 mL Ethanol *
- 1.8 mL Dichloromethane *

Hazards: *
- Ethyl acetate: Flammable (avoid flame)
- Dichloromethane: Flammable (avoid flame)
- Ethanol: Flammable (avoid flame)
- UV lamp: don't expose skin / sight
- Iodine is corosive: use under hood

~~* Ethyl Acetate is flammable while the other 3 compounds are~~

Hypothesis:

Because Silica gel (TLC plate) is polar, (highly polar), the more polar compounds will not move as far up the plate as the less polar compounds I predict that the order (from lowest R_f) the compounds will appear will be:

1) Caffeine 2.) Acetaminophen
3.) Aspirin 4) Salicylamide
5.) Ibuprofen. The lower the R_f value the less distance it travels. ∴ Caffeine will travel the least and will have the lowest R_f.

Caffeine is most polar because it contains two amine, two amide, and an alkene group

Procedure:

Obtain 10 x 6.6 cm TLC plate (silica) → Draw (with pencil) a line @ ~1 cm from bottom

From left to Right: spot ← acetaminophen, Aspirin, Caffeine Ibuprofen, and Salicylamide.
(use different capillarys)

↓

Pour mobile phase into 400 mL beaker until 0.5 cm deep

↓

Fold Filter paper 2' and place in beaker, cover with watch glass

Procedure Cont'd:

Place TLC plate in beaker, without touching filter

↓

When mobile phase reaches ~1cm from top of plate, use tweezers to remove plate and let dry on paper towel

↓

mark spots under UV lamp / Iodine chamber

↓

Use Ruler to measure distance from initial line to spot.

Record:

Observe

- Something dripped on my first TLC Plate
- Folded filter paper and added acetic acid
- Some spots smaller than others (aceta/aspirin)
- After UV, some spots big, some lanes had multiple spots

Calculate

Plate (1) Front: 7.5 cm

aceta: 3.0 cm

Aspirin: 4.0 cm

Caffein: 0.6 cm

2.6 cm?

Ibu: 5.5 cm

Salic: 4.8

Ref: 5, 3.8, 3, 0.6

Observe

Iodine
after ~~UV~~ acetam.
is darker under UV

- After Iodine, Aceta and Salicylamide turned yellow

Caffe

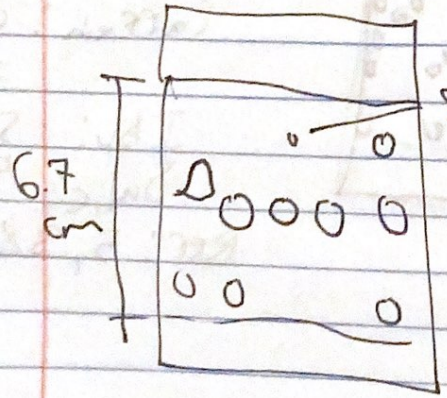
Observed two dots @ ~2.6cm for Caffeine. Maybe Contaminate

Use Mystery B

1	Anacin	3.7cm, 0.9	
2	Excedrin	0.9, ~~2.7~~ 3.0cm	(pinkish)
3	Tylenol	3.0cm	Small dot 5.4cm
4	Mystery B	3.0cm	
5	Ref	0.9, 3.0, 5.0	

- Excedrin had pinkish dot
- Anacin (3.7cm) had ghost shape

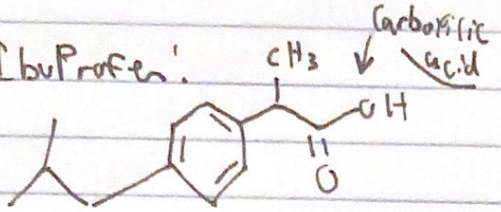
- Excedrin, Tylenol, Mystery turned yellow with Iodine @3cm
- Ref @ 5cm turned yellow



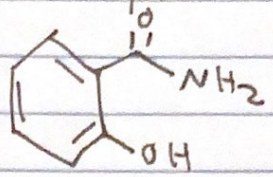
Mystery B looks like Tylenol

Spot	Distance (cm)	Color after UV	Color w/ Iodine
Acetaminophen	3.0	Purple	Yellow
Aspirin	4.0	Purple	White
Caffeine	0.6, 2.6	Purple	White
Ibuprofen	5.5	Purple	White
Salicylamide	4.8	Purple	Yellow
Ref	0.6, 3, 3.8, 5	Purple	Yellow/White
Anacin	0.9, 3.7	Purple	White
Excedrin	0.9, 3.0	Pink/Purple	Yellow/White
Tylenol	3.0	Purple	Yellow
Mystery B	3.0	Purple	Yellow
Ref	0.9, 3.0, 5.0	Purple	Yellow/White

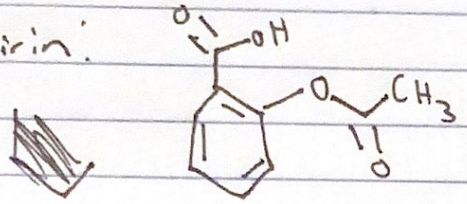
Ibuprofen:



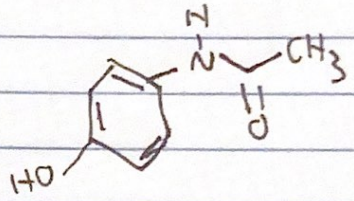
Salicylamide:



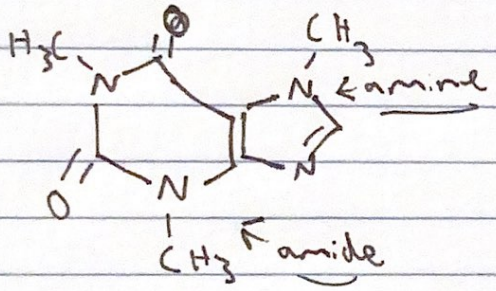
Aspirin:



Acetaminophen:



Caffeine:



Rf: Ibuprofen > Salicylamide > Aspirin > Acetaminophen > Caffeine

Josh Whitehead
u1069343
Chem 2315-001

Lab 1 “TLC” Conclusion

On my plate, Ibuprofen traveled the farthest, reaching 5.5 cm. Acetaminophen traveled 3.0 cm and aspirin traveled 4.0 cm. Due to differences in polarity, no two compounds traveled the same distance. It is important to record the Rf value of each compound because it is a ratio of how far the compound traveled to how far the mobile phase traveled so it is possible to compare to other plates.

The most polar compound out of those tested is caffeine because of its multiple amide and amine groups and an alkene group (see attached drawing). Caffeine is capable of forming dipole-dipole and London dispersion interactions with the silica. In contrast, the least polar is Ibuprofen because its carboxylic acid is its only polar group (see attached drawing). It is capable of London dispersion interactions with the silica. The results on the first plate support my hypothesis that caffeine is most polar and ibuprofen is least. Caffeine may have been impure because when held under the UV light, two spots appeared further up from the main spot in the same column.

UV light is needed to see how far the compounds traveled because when the mobile phase dries, it is not possible to discern where the spots ended. The UV light causes the spots to appear. Iodine was also used to help distinguish the spots. On my plate, after treating with iodine, acetaminophen and salicylamide turned yellow. This was helpful to align the compounds with the reference solution because some of the spots in that column turned yellow as well and those distances matched the distances of acetaminophen and salicylamide.

I saw two separate compounds in Anacin, two in Excedrin, and possibly two in Tylenol (the second spot appeared between two columns and it was very small). Comparing the Rf values from the individual compounds and in the over-the-counter drugs, it appears that Anacin contains caffeine and aspirin, Excedrin contains caffeine and acetaminophen, and Tylenol contains acetaminophen and possibly ibuprofen.

I used mystery “B” as my unknown solution and it appears to be Tylenol. Mystery “B” and Tylenol have the same Rf value and they both turned yellow after the iodine treatment. The second spot in the Tylenol column was also shared in the mystery column suggesting that they share that compound as well.

My plates turned out almost as expected except for two small miscellaneous dots in the caffeine column and one spot between the Tylenol and mystery columns in the second plate. I believe that the spot shared by Tylenol and the mystery column indicates a second compound that is shared between the two solutions that got too close so they formed a single spot. The extra spot in caffeine I believe is due to dripping something on my plate while spotting the initial line. In the reference for the second plate there was also one big spot where two smaller spots were expected. This may be due to the solution not having enough time for the two compounds to separate.

Josh Whitehead
u1069343
Chem 2315-001

Plate 1		
Compound	Rf*	Distance (cm)
Acetaminophen	0.40	3.0
Aspirin	0.53	4.0
Caffeine	0.08	0.6
Ibuprofen	0.73	5.5
Salicylamide	0.64	4.8

Plate 2		
Drug	Rf*	Distance (cm)
Anacin	0.13, 0.55	0.9, 3.7
Excedrin	0.13, 0.45	0.9, 3.0
Tylenol	0.45, 0.81	3.0, 5.2
Mystery B	0.45, 0.81	3.0, 5.2

*Rf value calculated by dividing distance traveled by spot by distance traveled by mobile phase. The distance traveled by mobile phase for plate 1 was 7.5 cm and for plate 2 was 6.7 cm.