CHM1321 Lab Report 1:

Thin Layer Chromatography

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Protocol

Please refer to pages 17-19 within the lab manual.
One modification is that we found it to be easier to draw the solvent line at the top before the TLC plates were placed within the jars.

Observations

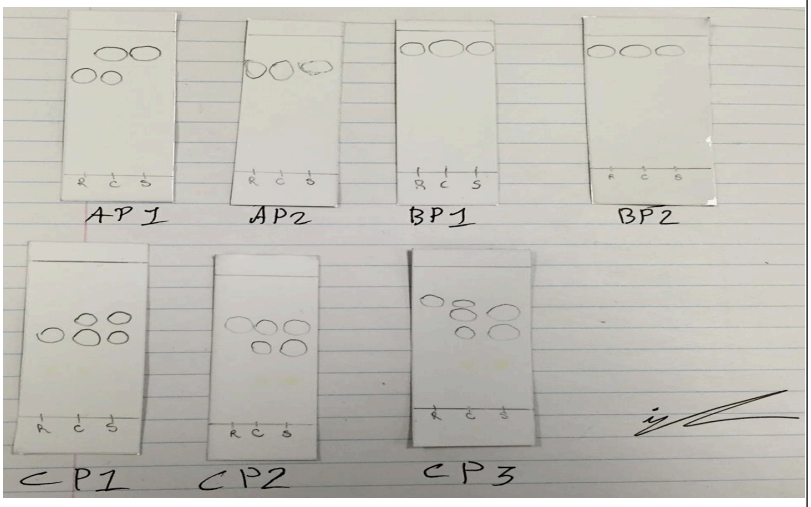
Unknown compound #36 appeared as white, fine crystal structures with no notable odor. When dissolved in Dichloromethane (clear liquid) the solution was also a clear liquid. This observation is the same for the appearance of the second reference solution, Benzophenone, and the resulting dissolved mixture.

TLC plates appeared white in front with a metal back. After sitting in the developing jar and put under a UV light, it was noted that the TLC plates appeared green, with black dotes, indicating how far up the solvent traveled on the TLC plate.

Unknown solution ZZ as well as reference solution C were both yellow in appearance. Reference solutions A and B were both clear in appearance. Reference solutions A, B, and C were odorless, while unknown solution ZZ's odor couldn't be observable because it had to remain within the fume hood.

TLC Plates

All the TLC Plates ***(Below is signed data sheet)***



Legend:

AP: TLC plate from part A

BP: TLC plate from part B

CP: TLC plate from part C

1: Plate 1

2: Plate 2

3: Plate 3

R: Reference Solution

C: Co-spot (Mix of S and R)

S: Solution

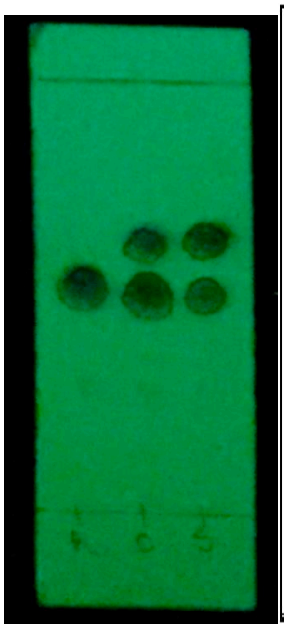
RF Table:

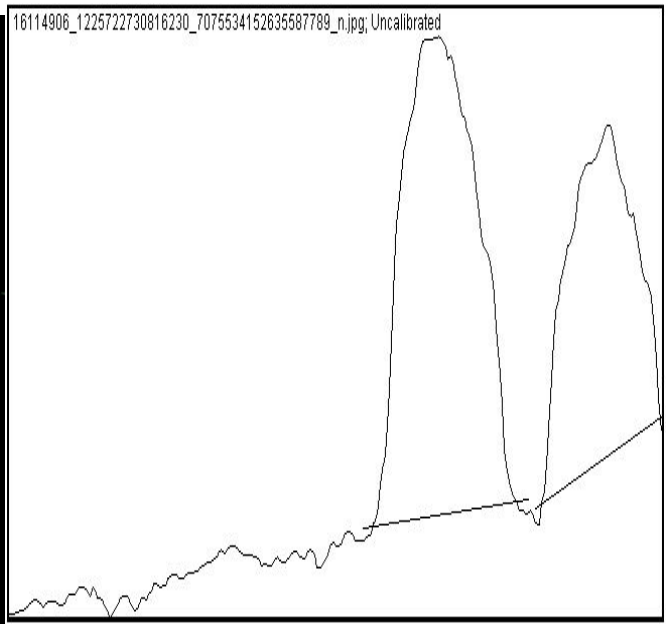
TLC Plate	Substance 36 RF	Dichloromethane RF	Benzophenone RF	Solution ZZ RF	Reference Solution A RF	Reference Solution B RF	Reference Solution C RF	Co-Spot RF
AP1	**0.81**	**0.67**						**0.67** **0.86**
AP2	**0.85**		**0.80**					**0.80**
BP1	**0.83**	**0.83**						**0.83**
BP2	**0.9**		**0.9**					**0.9**
CP1				**0.52** **0.63**	**0.52**			**0.51** **0.62**
CP2				**0.54** **0.66**		**0.67**		**0.54** **0.67**
CP3				**0.57** **0.7**			**0.75**	**0.57** **0.68** **0.76**

RF VALUE = DISTANCE FROM BASELINE(SOLUTE) / DISTANCE FROM BASELINE TO SOLVENT LINE

RF of Substance 36= 4.5cm/5.5cm = 0.818cm

Reference used in ImageJ(CP1), Graph from ImageJ using the "S"Colum(Second hill is first RF)





Calculations:

Absorbance of Meta = 23536
Absorbance of Ortha = 14445

%Absorbance (meta)= 23536 / 37981 x 100 = 61.9%
%Absorbance (ortha) = 100% - 61.9% = 38 .03%

y = %Absorbance(meta) = 61.9%
y = 1.7373 (x) + 0.31
61.9% = 1.7373 (x) + 0.31
61.59% = 1.7373 (x)
x = 35.45%
% mole (meta) = 35.45%
% mole (ortha) = 64.55%

Discussion

Errors/Justifications

- If you accidently touch the front of the TLC plate at any time one must start over.
- Labeling must be in pencil not pen, because pen ink smudges on the plate.
- Ensuring the solution line isn't too close to the bottom of the TLC plate, if the solutions on the plate mix with the solute in the jar than one must start over from the very beginning.
- After removing the TLC plate from the jar, one must let it dry before placing it under a UV light, because of you don't the TLC plate will be unusable and must be redone.
- Spotting must not be too small, as spots might not show up under the UV light.
- Spotting must not be too big, as spots might be too big and overlap other spots
- Spots irregular shape also made it difficult to find the middle for a proper value.
- Capillaries are fragile, if too much pressure is applied, then they will snap and one must retrieve a new one.
- Capillaries can get mixed up very easily. One could try to use the same end of the capillary to attempt to spot two different solutions (An explanation as to why plate AP1 and BP1 look different when they should look the same)
- Use of many capillaries and TLC plates were required because of the above possible errors.
- Using ImageJ for chart analysis can be risky because it might not have total accurate when analyzing the image used, resulting in skewed results.

Ameliorations

- Ensuring to have multiple test tubes, beakers, and jars as to have storage for all solutions being used and not wasting time repeatedly washing the same test tube 7 times
- Drawing in solvent lines before placing the plate within the jar, ensures more accuracy of how high up the solvent reached. One could miss it if its being done after as the TLC plate dries out very quickly.

Results

- The RF of Substance 36 and the RF of reference solution Benzophenone were almost identical, while the RF of reference solution Dichloromethane was higher than the other two. This means that **Substance 36 was composed of Benzophenone.**
- Comparing TLC plates CP 1, 2, and 3, it can be visually seen that CP1 and 2 are more similar than CP3, deducing that **Solution ZZ is composed of otha and meta Bromonitrobenzene.**
- Using imageJ and the calculations shown above, **unknown ZZ is composed of 35.45% meta-bromonitrobenzene and 64.55% otha-bromonitrobenzene.**

Questions 1&2

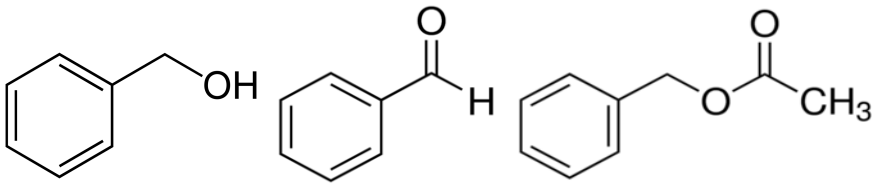
1) How does increasing the polarity of the solvent system affect the results of the TLC?

   Increasing the polarity in the solvent system will cause the polar solution to be bonding even more to the solvent, therefore causing the solution to travel faster up the TLC plate.
   If however the solution is nonpolar, increasing the polarity will cause the solution to travel slower up the TLC plate.

2) In the following sets of compounds, which would have the smallest RF on silica gel?
   a. Benzyl alcohol, benzaldehyde, benzyl acetate
   b. Aniline, naphthalene N,N-dimethylaniline
   c. Benzophenone, Biphenyl, Benzoic acid

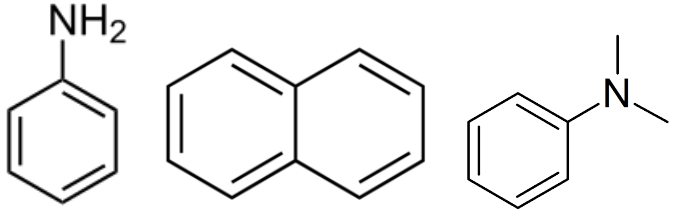
A) Benzyl Acetate is the least polar of the group, meaning it would travel the slowest up the TLC plate, inferring it would have the smallest RF

Benzyl alcohol Benzaldehyde Benzyl Acetate



B) Napthalene is the least polar of the group, meaning it would travel the slowest up the TLC plate, inferring it would have the smallest RF

Aniline Napthalene N,N-dimethylaniline



C) Biphenyl is the least polar of the group, meaning it would travel the slowest up the TLC plate, inferring it would have the smallest RF

Benzophenone Biphenyl Benzoic Acid