Introduction:

Thin Layer Chromatography (TLC) is a neat method used to identify organic compounds in mixtures and also to separate organic material; this allows the observation to see the completeness of the reaction. Thus, using this technique is useful. The TLC plates are made of aluminum on one side, and silica gel on the other. The silica gel is what will be used for the lab, to separate the organic compounds.

The purpose of part A of the experiment is to identify certain components of an unknown mixture using TLC plates. He migrated compounds could be detected by putting the TLC plate under a UV light, and the distance between the star and finish would give the Rf value. However, the purpose for part B is to observe how a different solvent is used to separate the same solution. Lastly, Part C, image J program is used to identify a new unknown mixture containing 2 different compounds, which then identifies the ratio of compounds.

Procedures:

Part A:

1. Two TLC plates were prepared by drawing a 1cm from the bottom of the plate
2. The plate was then divided into three parts: a) the Reference point b) co-spot and c) the sample.
3. Approximately 10mL of mixture 2:8 ethyl acetate hexane was put into the testing jar.
4. Ten grams of sample #7 was mixed with 2mL of dichloromethane in a test tube. This is the sample solution.
5. Approximately 2mL dichloromethyl and biphenyl were poured into 2 different test tubes.
6. A capillary was used to put the sample solution into the sample lane and co-spot lanes in both TLC plates that were prepared.
7. Dichloromethyl and biphenyl were both spotted in the reference lane in one of each TLC plates at the reference lane.
8. Both TLC plates were put into the developing jar.
9. After approximately 3-4 minutes, when the TLC plates eluted, the plates were taken out of the jar carefully ensuring they do not touch each other.
10. Both TLC plates were taken under the fume hood to be looked at under UV lights. And traced the points.

Part B:

1. Two new TLC plates were prepared.
2. The developing jar was empties, cleaned.
3. 10 mL of hexane was put in the jar.
4. A capillary was used to put the sample solution into the sample lane and co-spot lane in both TLC plates.
5. Dichloromethyl and biphenyl were both spotted in the reference lane in one of each TLC plates at the reference lane.
6. Both TLC plates were put into the developing jar.

7. After approximately 3-4 minutes, when the TLC plates eluted, the plates were taken out of the jar carefully ensuring they do not touch each other.
8. Both TLC plates were taken under the fume hood to be looked at under UV lights. And traced the points.

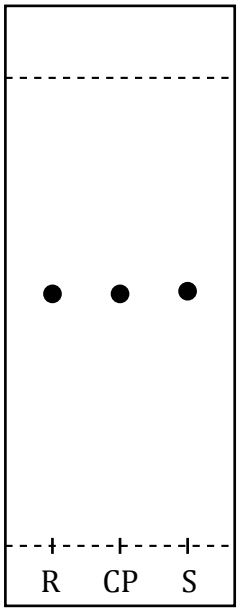
Part C:

1. three new TLC plates were prepared.
2. The developing jar was cleaned.
3. 10mL of 9:1 hexane was poured into the developing jar.
4. 2mL of solvent XX was put into a test tube.
5. Using the capillary, solvent XX was put in both TLC sample and co-spot of all three TLC plates.
6. Then one of each: p-bromonirtobenzen compounds, o-bromonitrobenzene, m-bromonitrobenzene was put in each TLC plate on the reference and co-spot lanes.
7. The TLC plates were viewed under the UV light to spot the differences, and then marked.

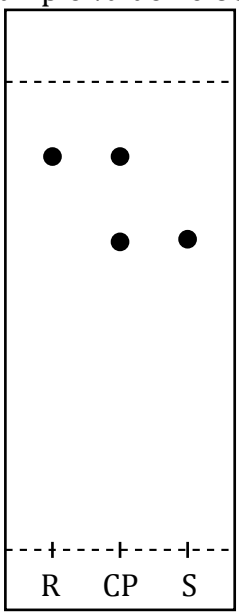
Observations:

Part A: solvent system 2:8 mixture of ethyl acetate and hexane

Reverence ***Benzophenone***
Rf Value= 0.56



Biphenyl
RF value= 0.84
Rf sample value= 0.66



Summary Table 1:

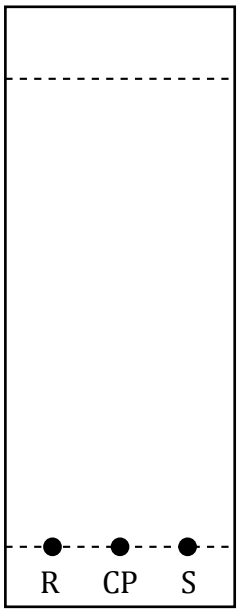
	Spots	Rf Value
Biphenyl	Reference Sample	0.84 0.66
Benzophenone	Reference Spot	0.56

The solvent in this part was 2:8 ethyl acetate mixtures The Rf Value was calculated by divining the distant traveled by the solvent by the distance total traveled by the solute. For example, biphenyl, the reference traveled 3.8cm and solute to a total of 4.5. Thus, (3.8/4.5)= 0.84. Since the biphenyl sample matched with the reference, it suggests that salt #7 is biphenyl compound.

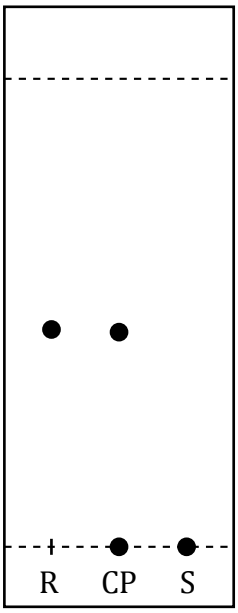
Part B:

Solvent system with pure Hexane solution

Reference: ***Benzophenone***
Rf value= 0

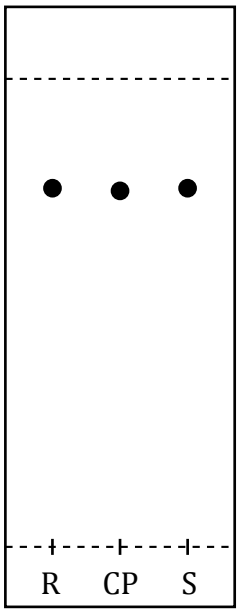


Reference: ***Biphenyl***
Rf Value=0.46
Spot Rf value= 0

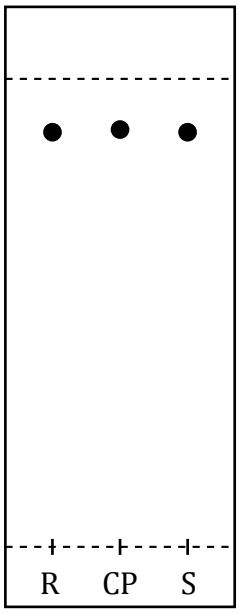


Solvent solution with pure Ethyl acetate

Reference ***Benzophenone***
Rf value= 0.77



Reference ***Biphenyl***
Rf value= 0.89



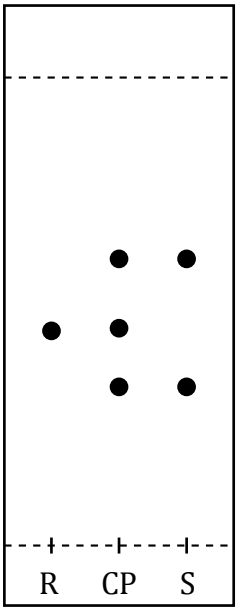
Summary Table 2:

Solvents		Spots	Rf Value
Hexane	Benzophenone	Reference	0
	Biphenyl	Reference Co-spot	0.46 0
Ethyl acetate	Benzophenone		0.77
	Biphenyl		0.89

Part C: Ratio of organic compound XX

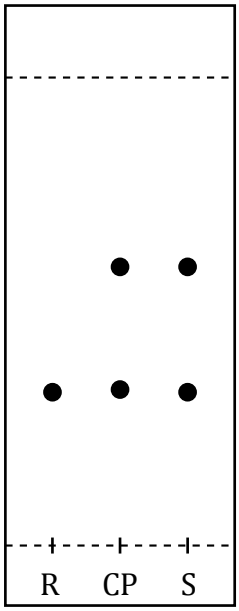
m-bromonitrobenzene

Reference= 0.46
Sample= 0.34 and 0.60



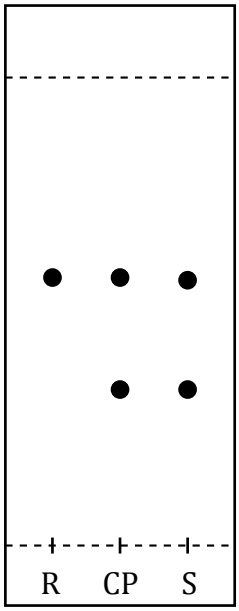
o-bromonitrobenzene

Reference= 0.33
Sample= 0.33 and 0.57



p- bromonitrobenzene

Reference= 0.57
Sample= 0.33 and 0.57



Summary Table 3:

Solvent		Spot	Rf Value
XX	m-bromonitrobenzene	Reference Co-spot Sample	0.46 0.60, 0.46, 0.34 0.60, 0.34
XX	o-bromonitrobenzene.	Reference Co-spot Sample	0.33 0.57, 0.33 0.57, 0.33
XX	p-bromonitrobenzene	Reference Co-spot Sample	0.57 0.57, 0.33 0.57, 0.33

Since both the p-bromonitrobenzene and 0-bromonitrobenze both had lower values than the m-bromonitrobenzene, it can suggest that both para and ortho are both components of the unknown sample XX.

Calculations:

Table 4: Peak % calculation

	Peak 2	**Peak 3**	**Total**	**% Peak 2**	**% Peak 3**
m-bromonitrobenzene	2359.024	1959.882	4318.906	54.6%	45.4%
o-bromonitrobenzene	6276.007	7366.401	13642.408	46%	53%
p-bromonitrobenzene	10337.472	5388.187	15725.659	65.7%	34.3%

Discussion:

The purpose of the lab was to determine certain components of an unknown mixture using TLC plates. In part A of the lab (using 2:8 ethyl acetate mixture), it was examined that salt #7 is crystalized biphenyl due to having the distance on the TLC plate higher than benzophenone, this suggests that the salt has the same polarity as biphenyl. Furthermore, comparing the polarities of Benzopheneone and Biphenyl to support this. Biphenyl has an Rf of 0.84 on the TLC plate while Benzophenone has an Rf of 0.56. This indicates that Biphenyl is less polar than Benzophenone. The less polar the compound is, the higher it goes up the TLC plate, which is the reason of its low Rf value. In addition, benzophenone contains oxygen in its molecule, which makes it polar, while biphenyl is pure C-H chains that are non-polar molecule.

In part B however, four TLC plates were prepared, 2 of which were to be tested with pure hexane, and the other 2 with pure ethyl acetate. Since ethyl acetate is more polar, it moves up the TLC plate, and also in a faster pace. While pure hexane is non-polar and does not interact with the silica gel of the TLC plate, which is why the Rf value of it is zero. Since Ethyl acetate is polar, for both compounds with biphenyl and benzophenone, have high Rf values of 0.89 and 0.77 respectively.

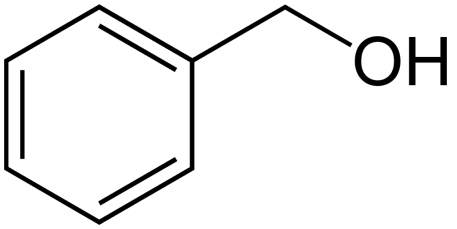
In part C the unknown compound that was used is XX. This compound produced spots in both ortho-bromonitrobenzene and para-bromonitrobenzene.

Pictures of the 3 TLC plates that were used in part C were scanned and analyzed using ImageJ program. The Co-sample lane was used to analyze the data for all three meta, ortho and para. By doing this, it is possible to calculate the percentage of each compound. This can be seen above at table 4.

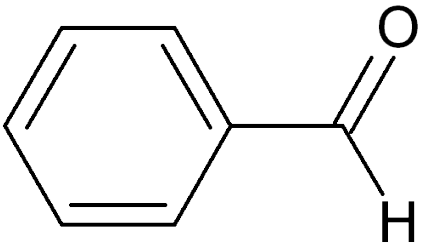
Analysis Questions:

1) Increasing the solvent polarity affects the TLC results by making the compound easier to attract than the silica gel. This would cause the compounds to move up faster on the TLC plate. In other words, high molarity breaks the attraction between the compounds and the silica gel on the TLC plate.

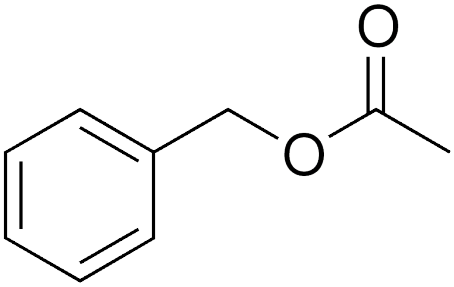
2) a. Alcohol groups are very polar compared to the other, thus explain the reason for them to move up the TLC farther up compared to others. Although they are all polar, benzyl alcohol since is it the most polar of the three. Not to mention that it has an OH group, the alcohol group that makes it the most polar (to support this also is the electronegativity between the O and H bond). This enables the group not only to accept H's but also donate. Benzalaldehyde is less polar because it does not have an OH bond making it less polar. Benzyl acetate, which its functional group only consists C-O bond and not any OHs, thus making it less polar.



Benzyl alcohol

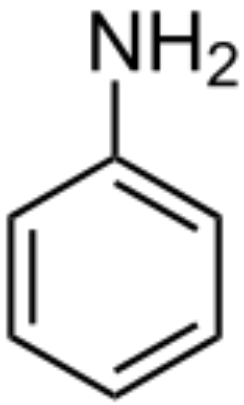


Benzaldehyde

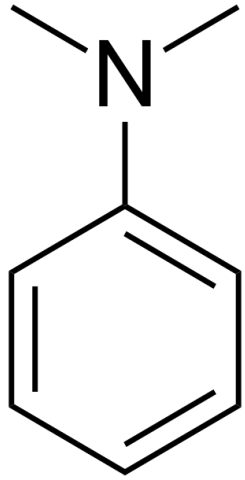


Benzyl Acetate

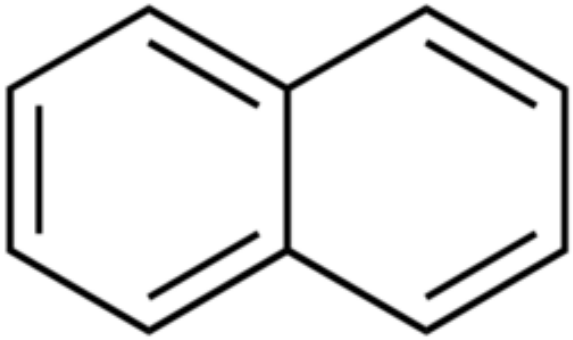
2b. With these 3 structures (structures are shown below the question), Aniline has a primary amine functional group where as N,N-dimethylaniline is a tertiary amine functional group. This makes Analine more polar, and also will be able to donate and receive H's. Not to mention that N-H bonds are much stronger than C-H bonds (N-H has hydrogen bonding). Thus, making Analine the smallest Rf value. Finally, naphthalene is the least polar due to having straight C-H bonds.



Analine

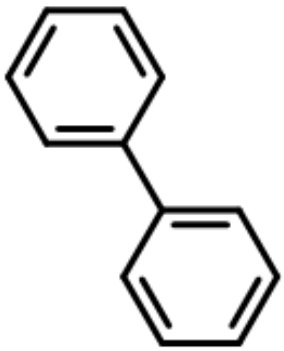


N,N-dimethylaniline

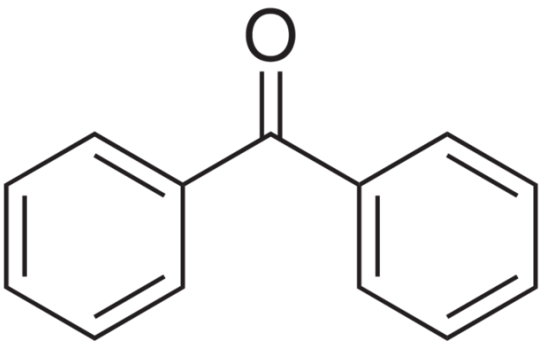


Nephthalene

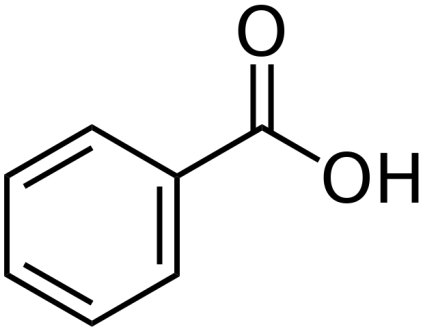
2c. Out of the 3 structures (shown below the question), the least polar one is biphenyl since it lacks a polar functional group. Since benzoic acid has an alcohol group it makes it more polar than the benzophenone which main functional group consist a C-O bond. Thus making it less polar than benzoic acid with has a more polar function group (the alcohol), and also the electronegativity values are higher between those two. The smaller Rf value will be that of benzoic acid.



Biphenyl



Benzophenone



Benzoic Acid