Introduction:

This lab is meant to show how the polarity of analytes affect the separation in thin layer chromatography.

Results:

Part A: Identifying the components of an unknown mixture using TLC.

Figure 1
Solvent: Ethyl Acetate 2:8 and Hexane
unknown: #82
unknown's weight: 0.0127 g
Distance travelled by solute:
Reference Benzophenone: 3.0 cm Sample: 3.9 cm
Distance travelled by solvent: 5.0 cm
R_F reference= 0.6
R_F sample: 0.78

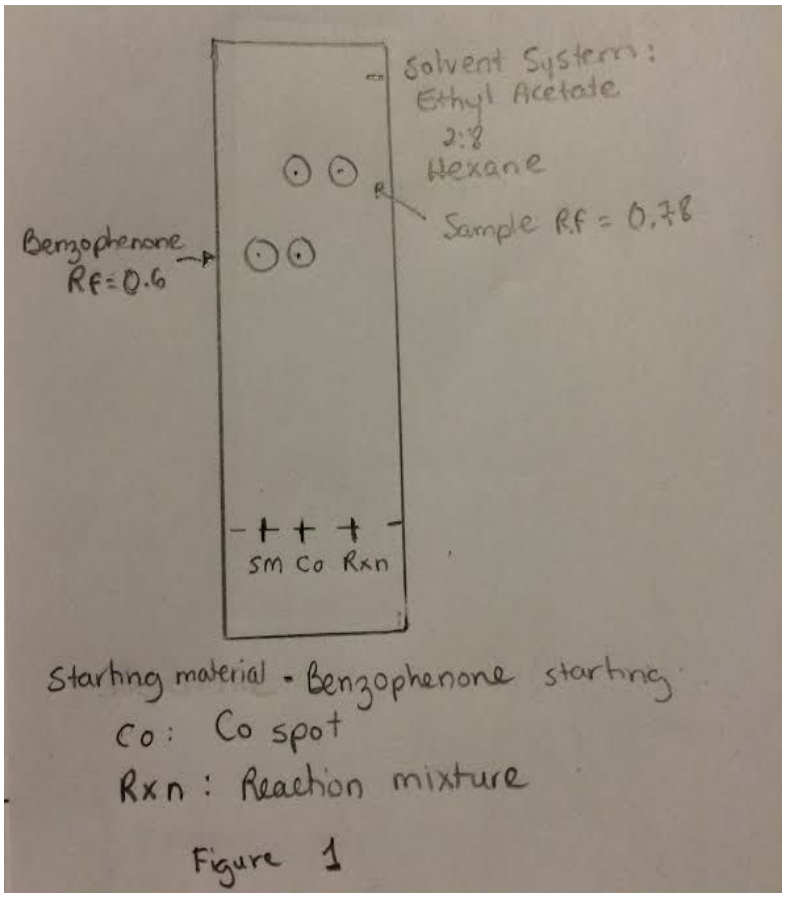


Figure 2:
Distance travelled by solute:
Reference Biphenyl: 4.1 cm Sample: 4.1 cm
Distance travelled by solvent: 5.5 cm
R_F reference= 0.75
R_F sample: 0.73

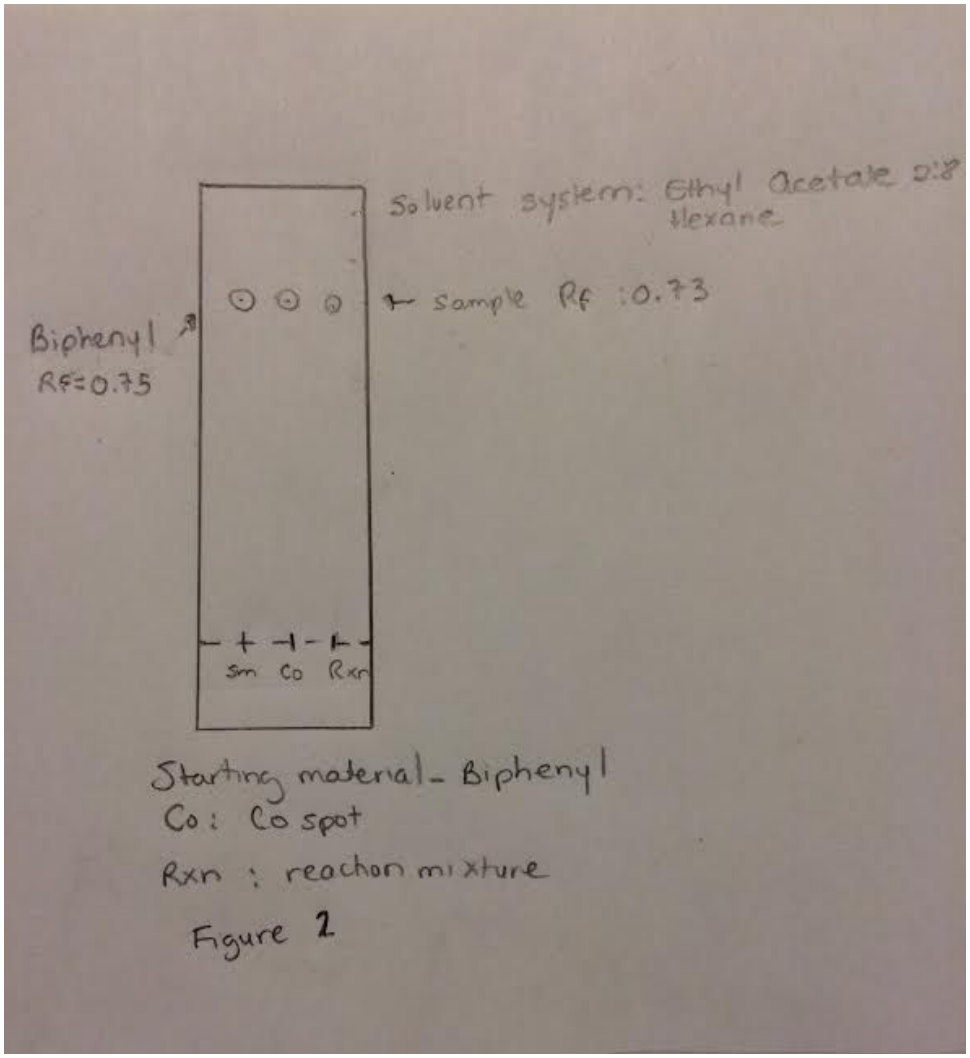


Figure 2

In the first part of the experiment we were given a sample of an unknown compound. Two TLC plates were used to analyze the sample: One plate had benzophenone and the other had biphenyl.

Part B_1: Effect of Solvent on TLC
Solvent: Ethyl Acetate

Figure 3
Distance travelled by solute:
Reference Benzophenone: 4.6 cm Sample: 4.7 cm

Distance travelled by solvent: 5.7 cm
R_F reference= 0.81
R_F sample: 0.82

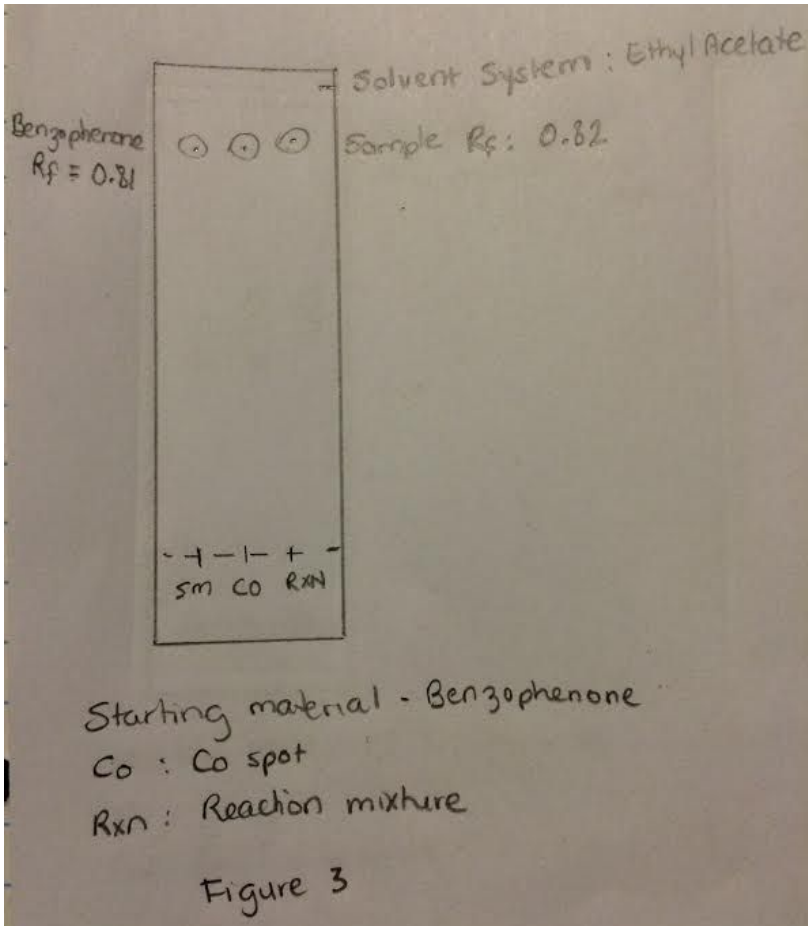
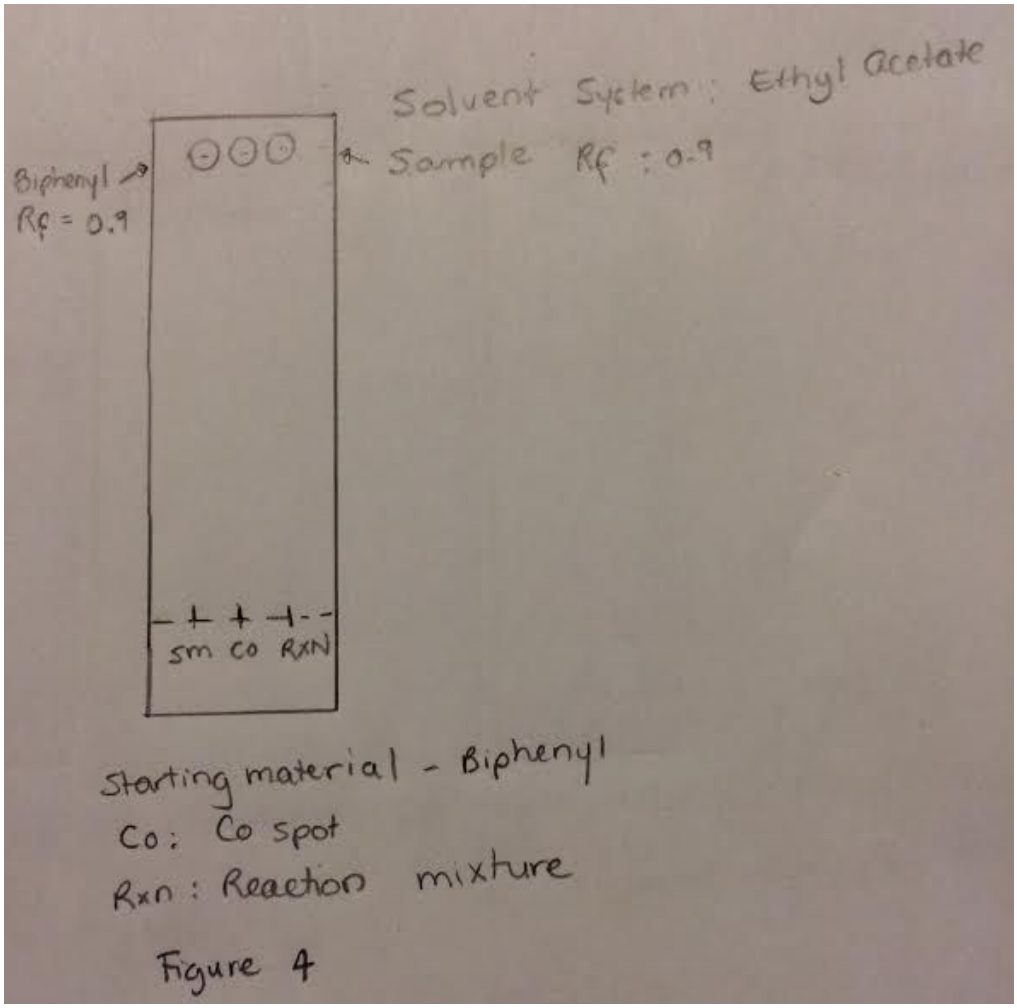


Figure 4
Distance travelled by solute:
Reference Biphenyl: 5.2 cm Sample: 5.2 cm
Distance travelled by solvent: 5.8 cm

R_F reference= 0.9
R_F sample: 0.9



In the second part of the experiment we used a different solvent: Ethyl Acetate and then used the same reference compounds as used in part A.

Part B_2:
Solvent: Hexane

Figure 5:

Distance travelled by solute:
Reference Benzophenone: 1 cm Sample: 3.7 cm
Distance travelled by solvent: 5 cm
R_F reference= 0.2
R_F sample: 0.74

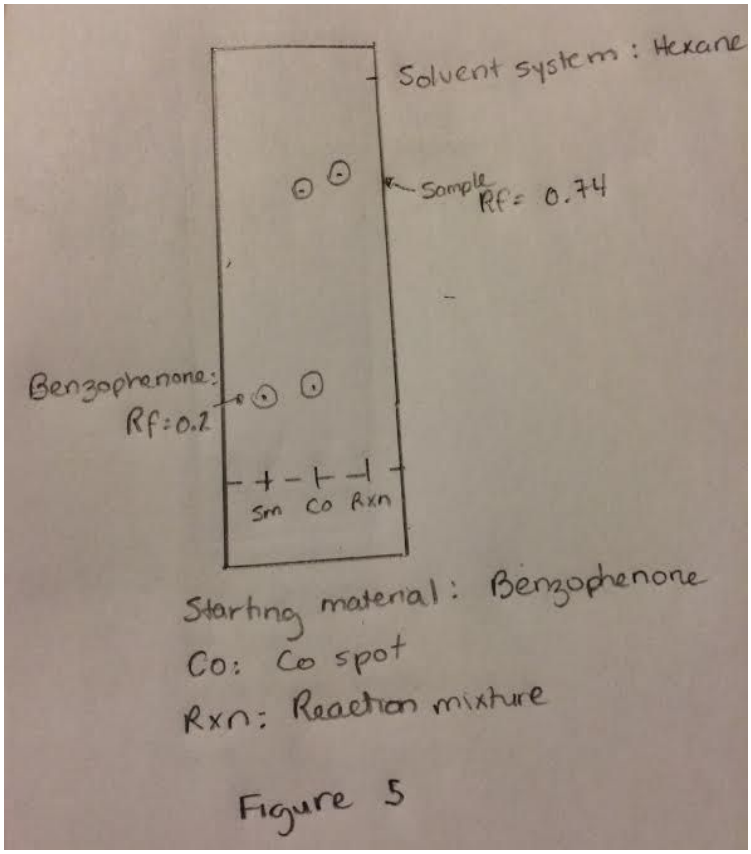
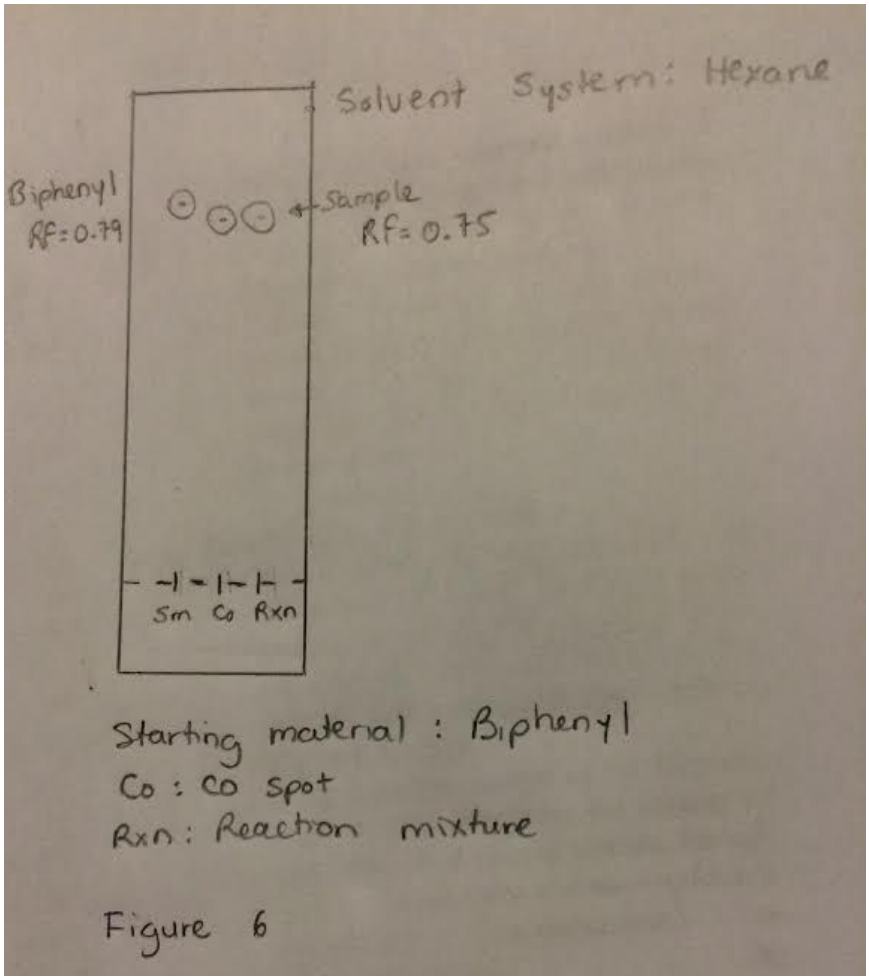


Figure 5

Figure 6
Distance travelled by solute:
Reference Biphenyl: 4.2 cm Sample: 4 cm
Distance travelled by solvent: 5.3 cm

R_F reference= 0.79
R_F sample: 0.75



Hexane is the solvent used for this section.

<u>Part C: Ratio of Compounds</u>
Unknown: YY
Solvent: 9:1 Hexanes: Ethyl Acetate

Figure 7
Reference compound: o-bromonitrobenzene
Ratio of compounds in the mixture using ImageJ:
m-bromonitrobenzene: (3587/ 3587 + 4294) x 100 = 45.5%
p-bromonitrobenzene: (4294/3587 + 4294) x 100 = 54.5%

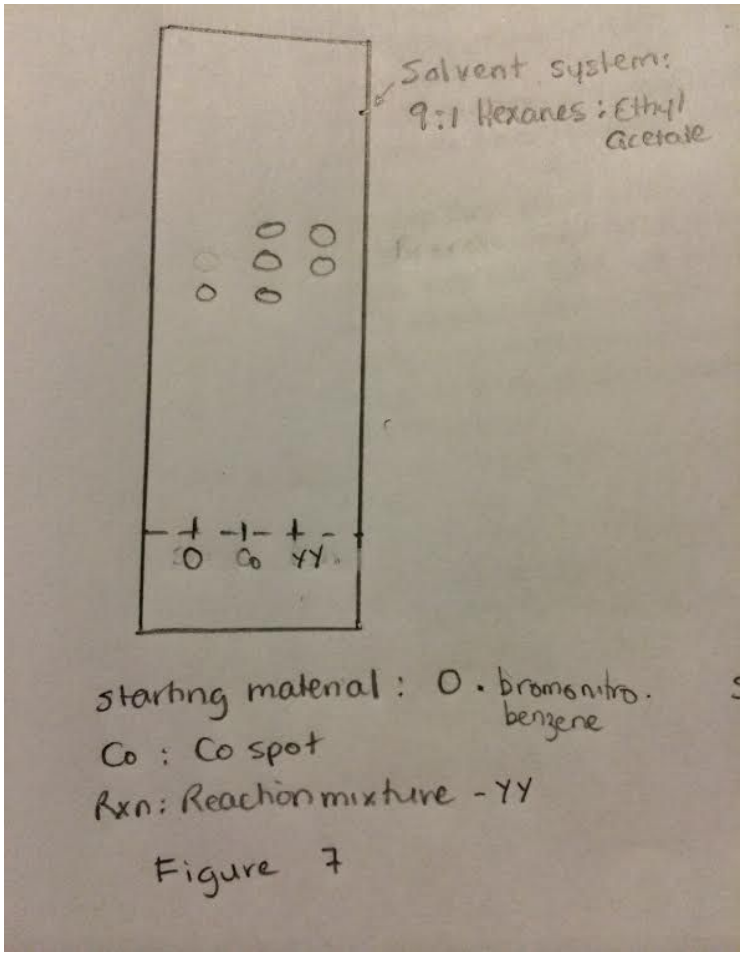


Figure 8
Reference compound: m-bromonitrobenzene
Ratio of compounds in the mixture using ImageJ:
m-bromonitrobenzene: (4014 / 4014 + 4148) x 100 = 49%
p-bromonitrobenzene: (4148 / 4014 + 4148) x 100 = 51%

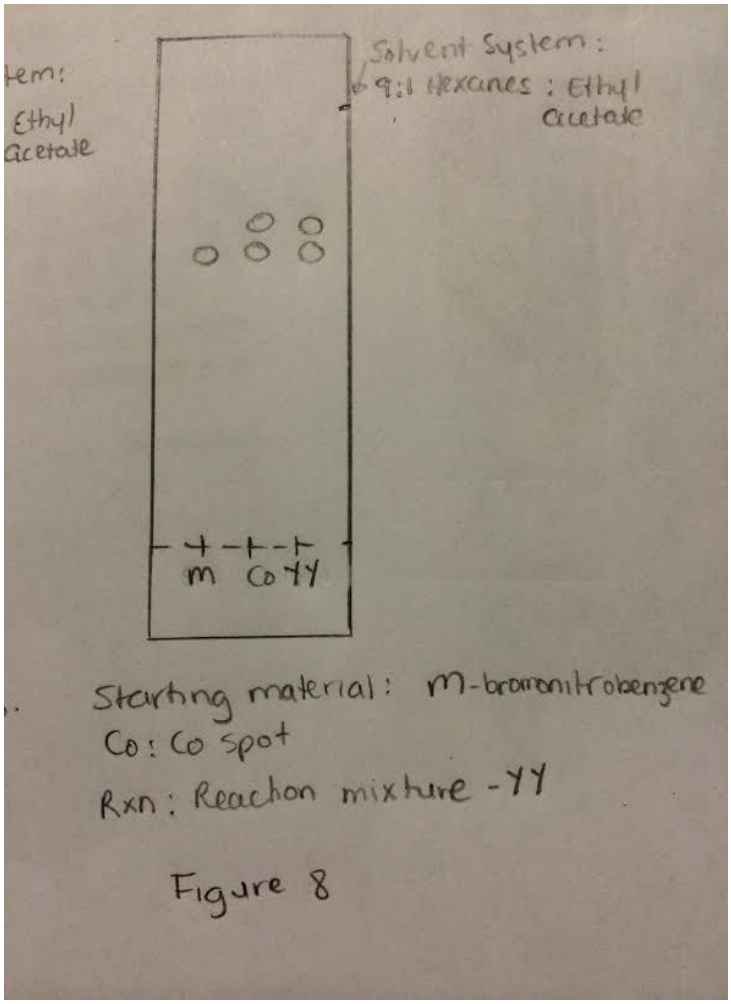


Figure 9
Reference compound: p-bromonitrobenzene
Ratio of compounds in the mixture Using ImageJ:
m-bromonitrobenzene: (6926/ 6926 + 8305) x 100 = 45%
p-bromonitrobenzene: (8305/ 6926 + 8305) x 100 = 55%

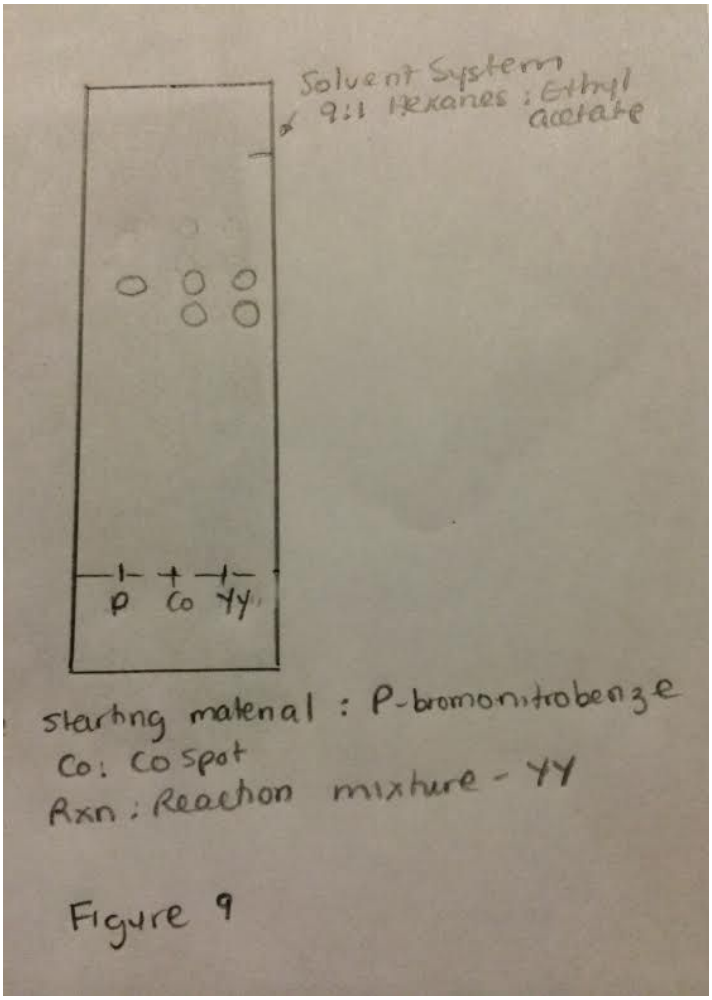


Figure 9

In section C of the experiment our solvent was 9:1 Hexanes: Ethyl Acetate. We were also assigned the unknown: YY. We needed to determine the identity of each component of our mixture by comparing TLCs with the following reference compounds: o-bromonitrobenzene, m-bromonitrobenzene, p-bromonitrobenzene. YY is m-bromonitrobenzene and p-bromonitrobenzene.

Calibration curves:
Calibration curve equation
47= 0.976x + 0.804
x=mol % = 47.3%
Average is 47%
Observations:
Part A

The solvent 2:8 Ethyl Acetate and Hexanes is clear and colourless. It has a strong “nail-polish remover” odour.
Dichloromethane is also clear, colourless. It also has a strong alcohol like smell.

Unknown #82- Crystallized, white, shiny surface, no odour.
benzophenone is clear, colourless, smells like nail polish remover. Biphenyl is clear, colourless, with a strong alcohol like odour.

Part B_1

Ethyl Acetate is clear, colourless and has a strong alcohol odour.

Part B_2

Hexane is clear, colourless, and has a strong odour (alcohol like)

Part C

9:1 Hexanes: Ethyl Acetate is clear colourless and has a strong alcohol like odour.
Unknown YY is clear, colourless with a strong alcohol like odour.
o-bromonitrobenzene: Transparent, yellow-did not notice odour
m-bromonitrobenzene: clear and colourless-did not notice odour
p-bromonitrobenzene: clear and colourless-did not notice odour

<u>Discussion:</u>

Part A:
Considering the R_F values Benzophenone R_F= 0.6 and Biphenyl R_F= 0.75, our sample was Biphenyl because its R_{Fs} were 0.78 and 0.73.

Part B: The separation in TLC is based on the affinity of analytes to silica. generally speaking, more polar compounds have higher affinity to the silica than to the solvent which is less polar than the silica. Therefore more polar solvents would cause analytes to migrate faster. In part B, we compared two solvents: Hexane and Ethyl Acetate.
Ethyl Acetate is more polar than Hexane because it has oxygens.
In hexane Biphenyl moved slower than in ethyl acetate with R_F 0.79 and 0.90 respectively.
The experiment for Benzophenone did not work. It seems that Biphenyl may have been used twice.
The sample compound moved faster in ethyl acetate than hexane with R_{Fs} 0.90 and 0.75 respectively.

Part C: According to the TLC results we are able to determine that the unknown YY is m-bromonitrobenzene and p-bromonitrobenzene because when you put the standard of o-bromonitrobenzene, you see that there is no o-bromonitrobenzene in the sample. Also, according to TLC results o-bromonitrobenzene is the most polar, following with m-bromonitrobenzene, and then p-bromonitrobenzene which is the least polar. o-bromonitrobenzene is the most polar because it was the slowest when migrating.

Interpretation of each TLC:
Part A:
Plate 1: Shows that our sample R_F = 0.78 has higher R_F value than Benzophenone R_F = 0.6, meaning that it is less polar.

Plate 2: The Biphenyl R_F= 0.75 has approximately the same R_F value than the sample- R_F = 0.73 meaning that they have the same polarity.

Part B:
Plate 3: Benzophenone has an R_F = to 0.81 and has approximately the same R_F as the sample (R_F= 0.82) This was likely an error because the other two experiments showed that the sample is less polar than benzophenone.

Plate 4: Biphenyl has a R_F of 0.9 and so does the sample which indicates the same polarity.

Plate 5: Benzophenone R_F (0.2) is much smaller than the sample R_F (0.74) meaning that Benzophenone is more polar than the sample.

Plate 6: Biphenyl R_F (0.79) is approximately the same R_F (0.75) as the sample which means that they have the same polarity.

Part C:

Plate 7:- YY mixture is added, but we know that there is no o-bromonitrobenzene in the sample when you put the standard of o-bromonitrobenzene, because you don't see it in on the sample side of the TLC. o-bromonitrobenzene is the most polar because it migrated the slowest.

Plate 8: m-bromonitrobenzene- YY mixture is added and we know that there is m-bromonitrobenzene in the sample because you could see it on the TLC. m-bromonitrobenzene is more polar than p-bromonitrobenzene but less polar than o-bromonitrobenzene.

Plate 9: p-bromonitrobenzene- YY mixture is added and we know that there is p-bromonitrobenzene in the sample because you could see it on the TLC. p-bromonitrobenzene is the least polar of the three reference compounds.

Conclusion:
Part A: The sample was Biphenyl because the R_{Fs} were 0.78 and 0.73.
Part B: Ethyl Acetate is more polar than Hexane because it has oxygens.
In hexane Biphenyl moved slower than in ethyl acetate with R_{Fs} 0.79 and 0.90 respectively.
The experiment for Benzophenone did not work. It seems that Biphenyl may have been used twice.
The sample compound moved faster in ethyl acetate than hexane with R_{Fs} 0.90 and 0.75 respectively.
Part C: Unknown YY is m-bromonitrobenzene and p-bromonitrobenzene, o-bromonitrobenzene is the most polar because it was the slowest migrating.

Questions:

1) The separation in TLC is based on the affinity of analytes to silica. generally speaking, more polar compounds have higher affinity to the silica than to the solvent which is less polar than the silica. Therefore more polar solvents would cause analytes to migrate faster.

2) Answers first, pictures to follow:

a.Benzyl alcohol has the smallest R_F- the OH group can readily form hydrogen bonds and is slightly acidic.

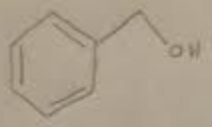
b. Aniline has the smallest R_F - because the amino group is the most polar and can form hydrogen bonds with silica.

c. Benzoic acid has the smallest R_F - because the carboxylic group is highly polar, and the hydrogen is acidic.

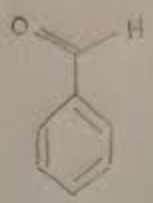
Figure 10
Pictures of Compounds for question #2

2

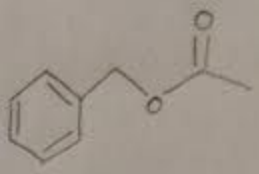
A) Benzyl alcohol



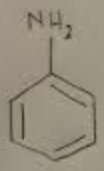
Benzaldehyde



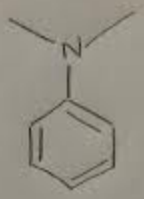
Benzyl Acetate



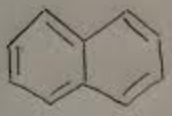
B) Aniline



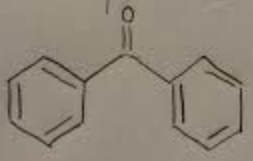
N,N-dimenthylaniline



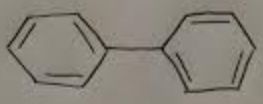
Naphtalene



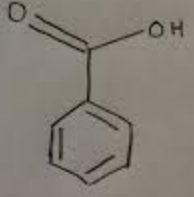
C) Benzophenone



Biphenyl



Benzoic acid



Raw Data:

Raw Data PART A

10 mL of a 2:8 mixture of ethyl acetate and hexanes - strong odour
clear + colourless

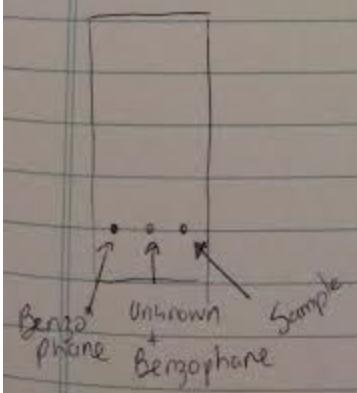
1-2 mL of dichloromethane - clear + colourless
smell is strong / alcohol like.

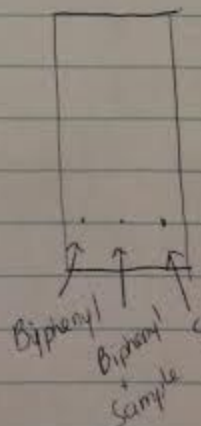
unknown #82
0.0127g
white
crystallized
shiny surface
(No smell

Biphenyl 2 mL
alcohol smell
clear - colourless

Benzophenone
smells like nail polish
clear + colourless
approximatel 2 mL.

(4) R(4)





in section A the Sample + Benzophenone ended up moving at different speed, & did not reach the same height

PART B
effect of Solvent on TLC

ethyl acetate
clear - colourless
strong odour
10 mL

Same thing as part A with new solvent
- Every Remains same colour / Clear colourless
Smell is still strong.

Benzophenone Biphenyl
circles approximately same spot but quite high.

hexane
clear, colourless, alcohol / nail polish remover smell.

Part C - (YY) clear / colourless / smell ?
9:1 Hexanes
clear colourless / strong odour

o → transparent yellow
m → clear + colourless
p → clear + colourless