Experiment 1: Thin Layer Chromatography

Date: January 22, 2019

Procedure:

Procedure as described in the lab manual.

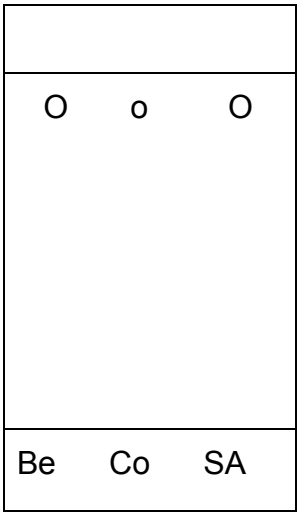
TLC & R_F Values

Key Equations in this section are: $R_F = \frac{\text{Distance travelled by the compound}}{\text{Distance travelled by the Solvent}}$

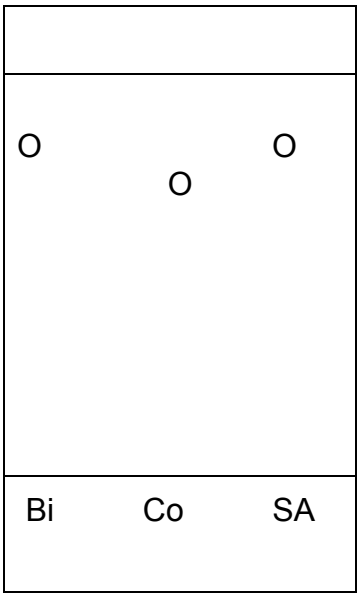
- *The experimenter was having technical difficulties importing the images into the file and had to use a screen shot for the TLC plates.*

Solvent system: EtoAc Hexanes: 2:8

Unknown Rf: 0.68 & Benzophenone Rf 0.71

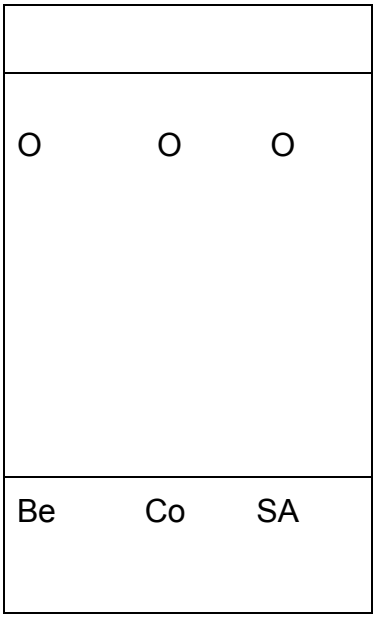


Be: Benzephone Rf: 0.71
CO: cospot
SA: Unknown 78# Rf: 0.68

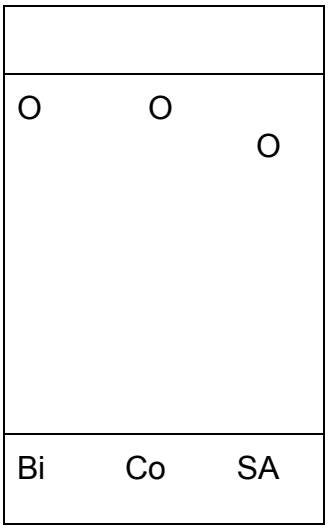


Bi: Biphenyl Rf: 0.70
Co: co-spot Rf: 0.46
SA: unknown Rf: 0.52

Solvent System: EtoAc

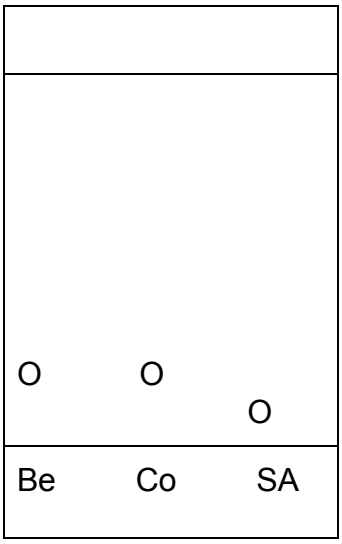


Be: Benzophenone Rf: 0.92
Co: Co spot
SA: unknown 78 Rf: 0.90



Bi: Biphenyl Rf: 089
Co: co spot
SA: unknown #78 Rf: 0.83

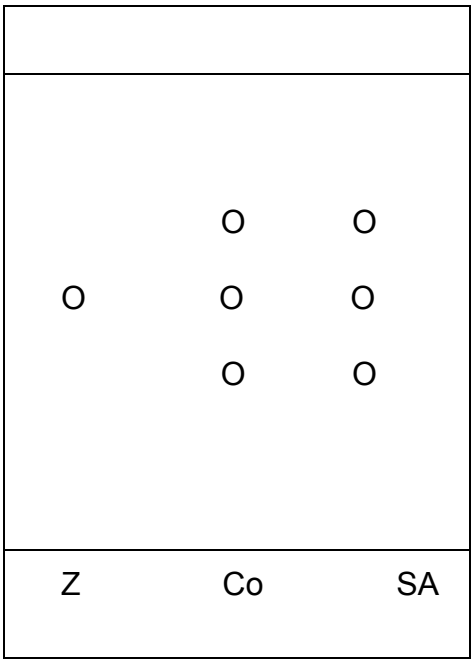
Solvent system: Hexanes



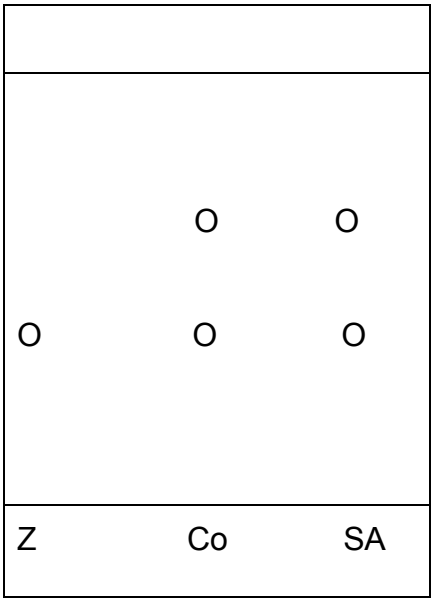
Be: Benzophenone Rf: 0.020

Co: co spot
SA: # unknown 78 Rf: 0.10

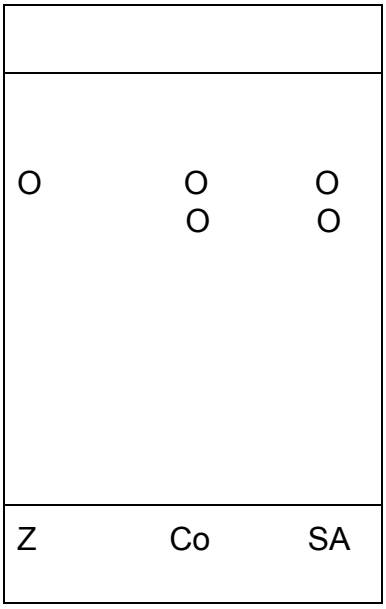
TLC'S for part C (solvent system Hexanes: Ethyl Acetate: 9:1)



Z: m-bromonitrobenze
CO: co spot
SA: unknown (XX)
In descending order: Dot 1 : 0.64, Dot 2: 048, Dot 3: 0.37

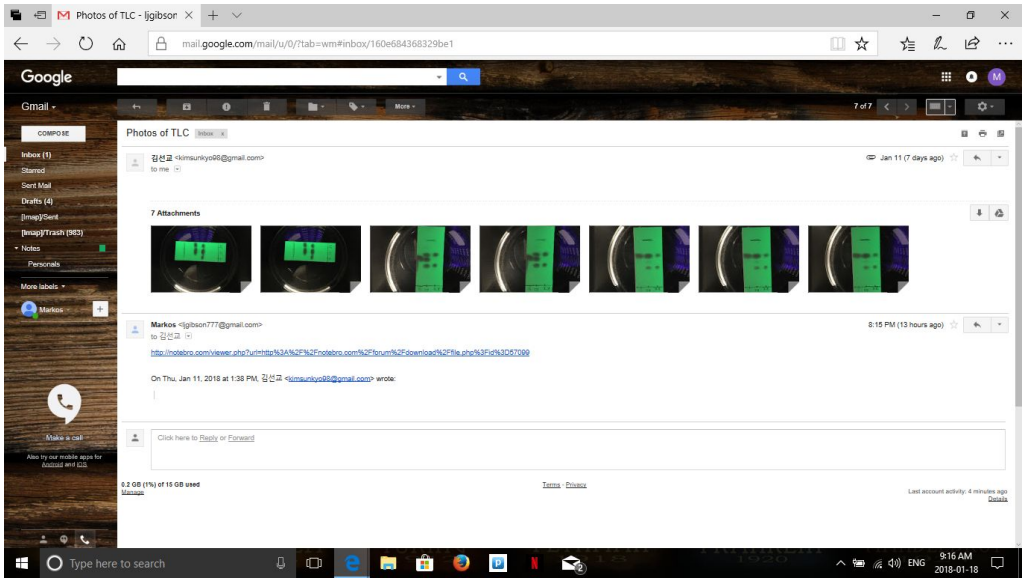


Z: O-bromonitrobenzene
Co: co spot
SA: Unknown (XX)
In descending order: Dot 1: 0.57, Dot 2: 0.34



Z: P Bromonitrobenzene
Co: Co spot
SA: unknown (XX)
In descending order: Dot 1: 0.61, Dot 2: 0.4

Photos of TLC'S



Procedure:

As described in the lab manual (CHM 1321 Organic Chemistry Laboratory, Dr. Rashmi Venkateswaren, 2018, Exp. 1, p. 16-18)

Results and Observations

Part A

	Benzophenone	*Biphenyl*
Unknown # 78	- *White granular crystals (gritty)* - *No detectable odor* - *Dissolved in 1.2 ml of dichloromethane (crystals dissolved to form a clear colourless solution that had a mild sweet odor)*	
Solvent system	*ETOAc: Hexanes: 2:8*	
Observations	- *Clear and colourless* - *Faint smell of balsamic* - *Rose swiftly*	- *Clear and colourless* - *Faint smell of balsamic* - *Rose swiftly*

Part B

Unknown # 78	*Benzophenone*	Biphenyl
Solvent System	ETOAc - Colourless liquid strong odor similar to glue or nail polish.	
Observations	- *Clear and colourless* - *Rose more swiftly than part A*	

Unknown # 78	*Benzophenone*	*Biphenyl*
Solvent System	Hexanes - Clear, colourless and very pungent odor.	
Observations	- *In Comparison to previous results, (TLC) was fairly faster at rising*	

Part C

m-bromonitrobenzene		*o-bromonitrobenzene*	*p-bromonitrobenzene*
Unknown	*XX* - *Mixture is yellow/green in colour and translucent*		
Solvent System	- *Hexane: Ethyl Acetate: 9:1* - *Clear, Colourless and pungent and acidic odor*		
Observations	*-Clear and colourless* *-rose swiftly*	- *Clear and watery yellow in colour* - *Mild speed in rising*	- *Clear and colourless* - *Very slow riser*

Calculation Part C (data analysis)

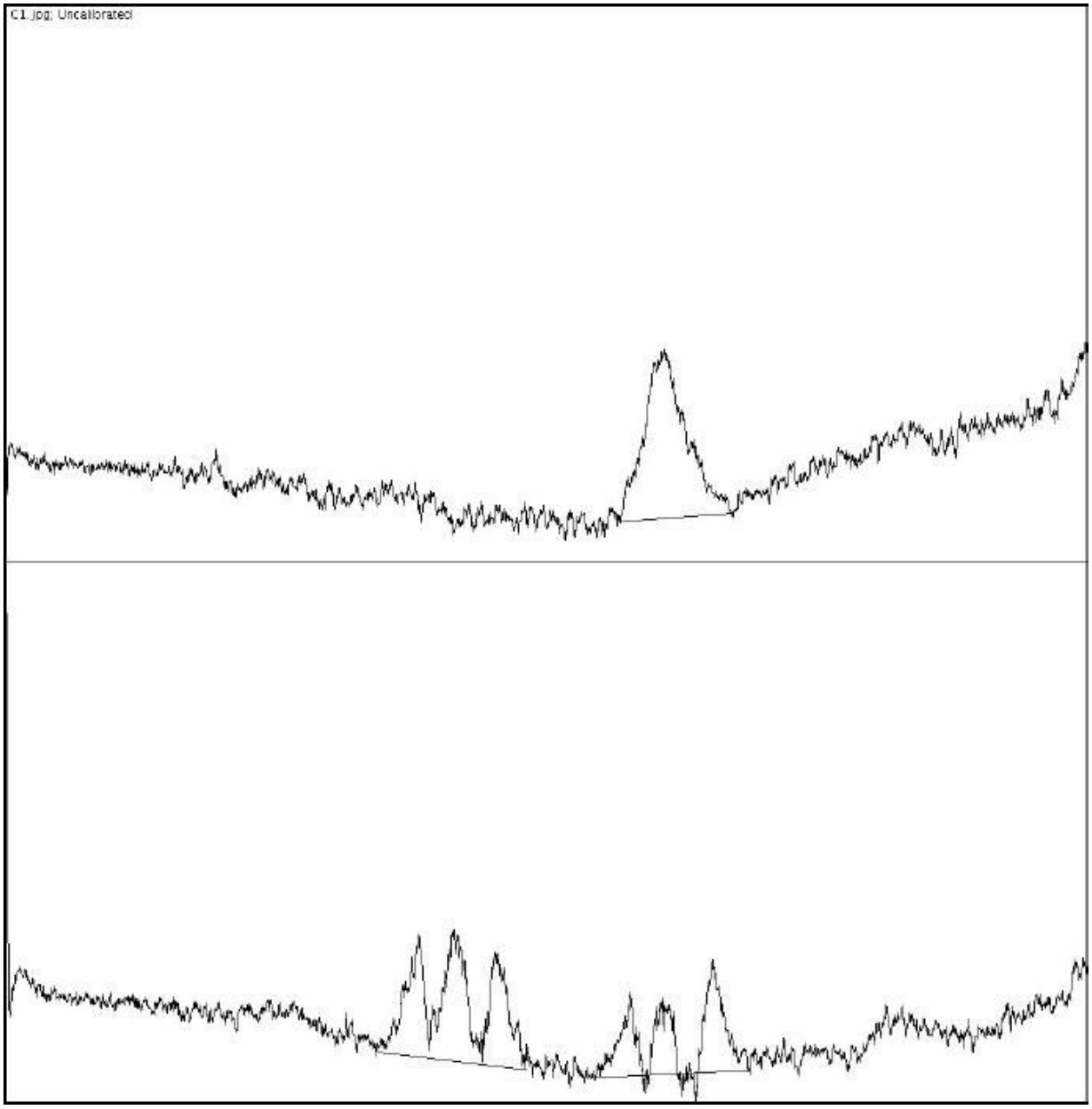


*Figure 1- is an illustration of the O-bromonitrobenzene vs. our TLC with the (XX) compound the calculations can be found within the table below. (**First peaks are not shown in each in figures 1&2 for clarity)***

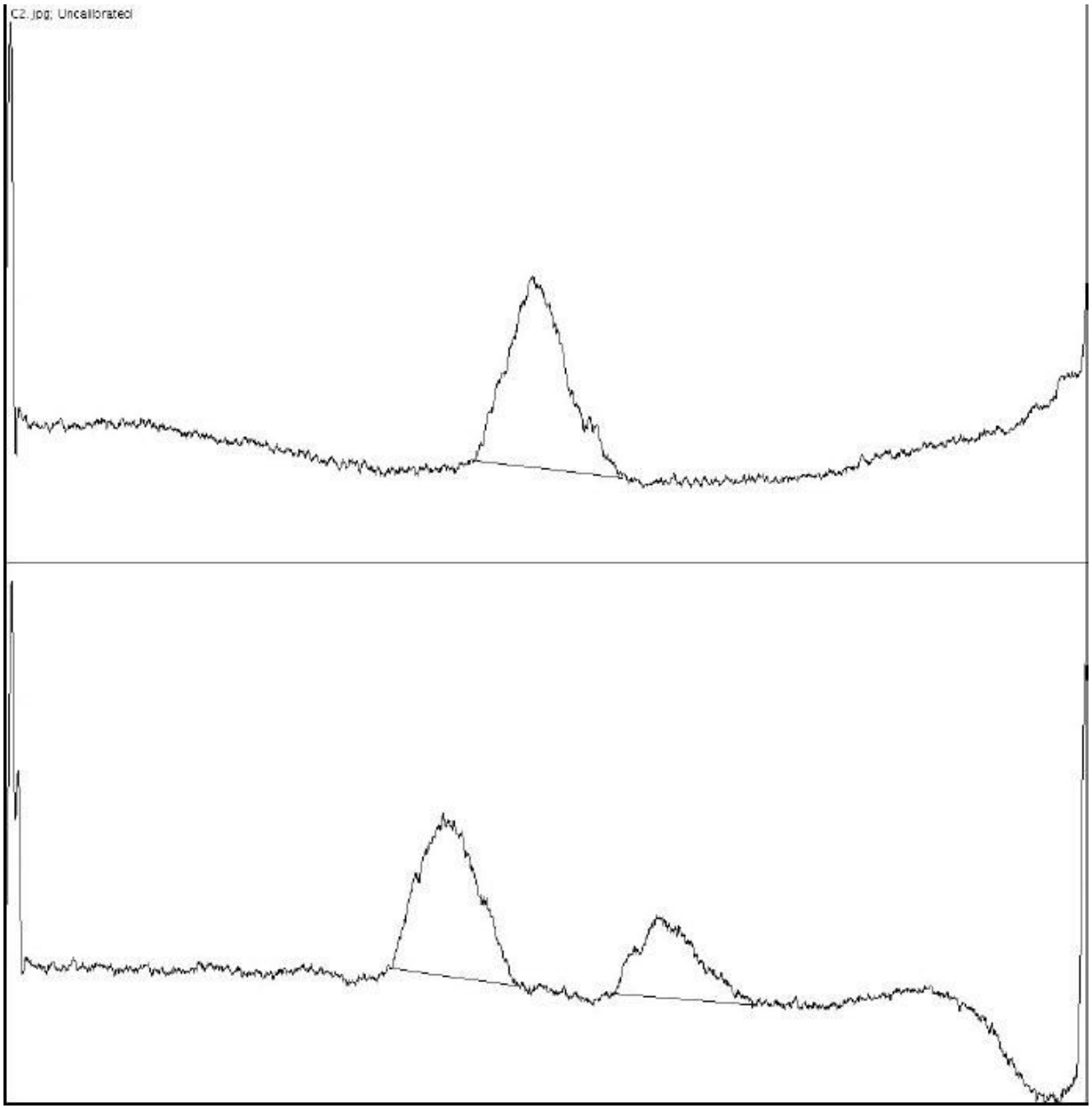


Figure 2- is an illustration of the m-bromonitrobenzene vs. our (XX) compound

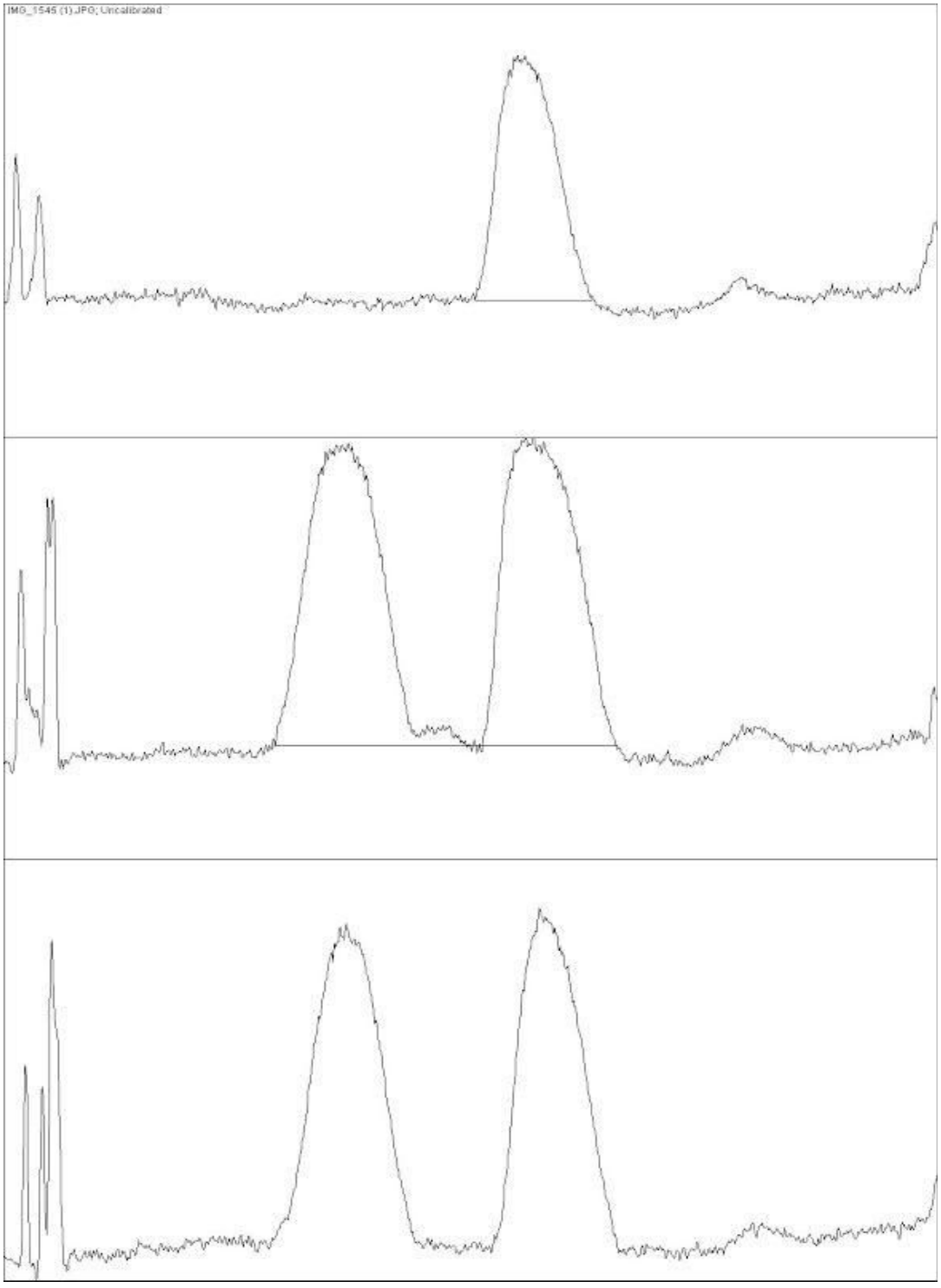


Figure 3 - is an illustration of p-bromonitrobenzene vs. our (XX) compound

Key equations used:

$$\%\text{Peak 2...} = \frac{\text{Area of Peak 2}}{\text{Area of Peak 2 + Area of Peak 3}} \text{ (multiplied by 100)}$$

Solutions	Area of peak 1	Area of Peak 2	Area of Peak 3
O-bromonitrobenzene	7916.439	8121.794	5019.924
m-bromonitrobenzene	12211.108	10737.165	5371.823
p-bromonitrobenzene	37110.543	18101.356	21101.012

Determining peak % of O-bromonitrobenzene

% Peak 2: 8121.794/ (8121.794 +5019.924) x 100%

= 61.8%

% Peak 3: 5019.924/ (8121.794+ 5019.924) x 100%

= 38.2%

Determining the peak % of M- bromonitrobenzene

%Peak 2: 10737.165/ (10737.165 + 5371.823)x 100%

= 66.65%

%Peak 3: 5371.823/(5371.823 + 10737.165)x 100%

= 33.34%

Determining the peak % of P- bromonitrobenzene

% Peak 2: 18101.356/ (18101.356 + 21101.012)x 100%

= 46.17%

% Peak 3: 21101.012/ (21101.012+ 18101.356)x 100%

= 53.82%

Average % composition of para-bromonitrobenzene

= [61.8+46.17] / 2

= 53.98%

Average % composition of Ortho- bromonitrobenzene

= [53.82 + 38.82] /2

= 46.32%

Key equation 2:

y= 1.0114X- 2.0208 (Para- bromonitrobenzene mixture)

53.98 = 1.0114X - 2.0208

X= 55.36 Jmol% of para-bromonitrobenzene

Jmol% of Ortho Bromonitrobenzene = 100 - 55.36

= 44.64 %

Errors

- Keeping the plate in the developing jar for too long, which can cause molecules will travel too far along the plate and won't show separation
- Keeping the plate in the developing jar for not enough time, which causes molecules to travel in different lengths and inconsistently, to solve this use a consistent time frame for plate developing
- Touching the TLC which can cross contaminate it with other molecules on hands, use prongs while holding TLC plates
- Cross-contamination of the capillaries used in spotting the TLC plates, make sure that all capillaries used are unique to a certain substance, or use multiple fresh capillaries

Discussion

Part A

- Biphenyl is non-polar compared to benzophenone which could have an affect on its Rf value
- Biphenyl is therefore more likely to stick to the silica gel which may increase its Rf value
- The data in part A suggests this.

Part B

- Ethyl Acetate can be seen as a polar molecule as it has a ketone and lone pairs and oxygen can bond to the polar silica gel
- Hexane is nonpolar and without symmetry and has no functional groups to bond with.
- As hexane is nonpolar and silica gel is polar overcoming dipole interactions would be difficult
- Due to the above factors, ethyl acetate may have a smaller Rf value.

Part C

- The symmetry of the graphs suggest the presence of both P-bromonitrobenzene and m-bromonitrobenzene in a fixed ratio
- It is also important to note that functional groups can affect polarity

Questions

1. **Why is it important to make co-spots last?**

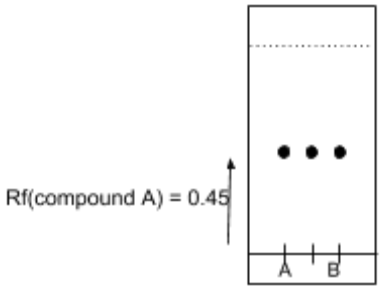
It is very important to place the co-spots onto the TLC plates last so that the reference and sample spots are not contaminated.

2. **How does increasing the polarity of the solvent system affect the results of a TLC?**

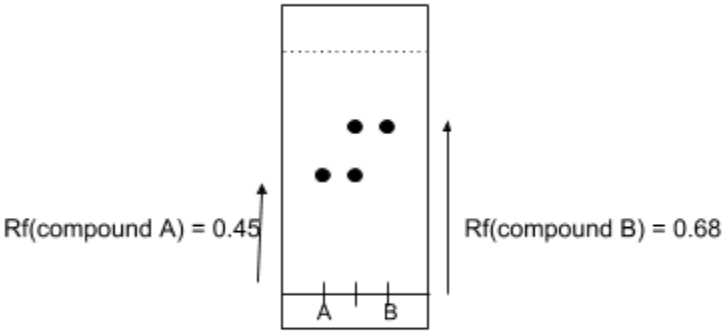
Increasing the polarity of the solvent causes an interaction between the intermolecular forces between the silica gel and compounds to be interrupted, which therefore allows them to travel farther.

3. **A student is monitoring a reaction using TLC. The chemical reaction converts compound A into compound B, and she uses molecule A as her reference. Compound A has an Rf value of 0.45; while compound B has an Rf of 0.68 in the solvent system being used.**
    a. **Draw a picture of her TLC plate at the beginning of the reaction.**

b. Draw a picture of her TLC plate after 50% completion (50% of the A molecules have been converted to B molecules).



c. Draw a picture of her TLC plate at the end of the reaction (all of the A molecules have been converted to B molecules).



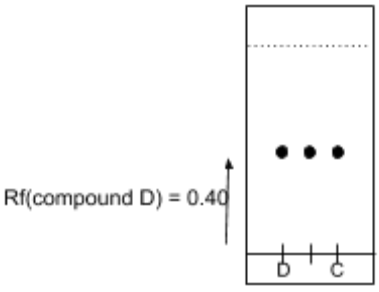
d. Why is it better to use a sample of molecule A rather than molecule B to follow the reaction?

A sample of molecule A has a lower R_f value. What that does it cause the molecules to not travel as far on the TLC plate as molecule B. An explanation for this interaction could be that molecule A is actually more polar than molecule B. This causes molecule

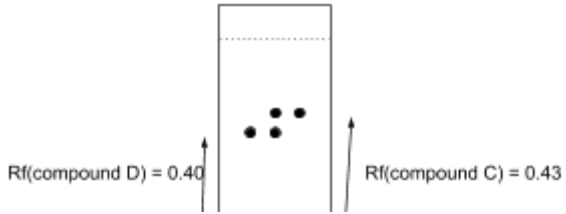
A samples to interact with the silica gel on the TLC plate more through hydrogen bonding. The opposite is true for molecule B, that due to its higher Since A has a lower R_f value it means that the molecules will not travel as far up the plate as molecule B. This could be due to the fact that molecule A is more polar than molecule B, therefore interacting more between the silica molecules through hydrogen bonding. Since molecule B has a higher R_f it will travel further up the plate, due to its very probable lower polarity level.

4. **A student is monitoring a reaction involving compounds C and D using TLC. Compound C has an Rf value of 0.43; compound D has an Rf of 0.40.**
    a. **Draw a picture of the TLC plate at the beginning of the reaction.**

    b. **Draw a picture of the TLC plate after 50% completion (50% of the C molecules have been converted to D molecules).**



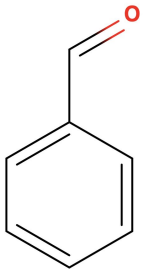
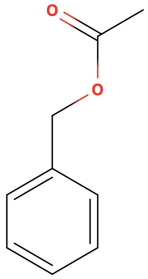
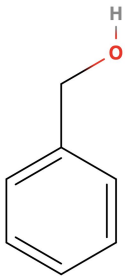
    c. **Draw a picture of the TLC plate at the end of the reaction (all of the C molecules have been converted to D molecules).**

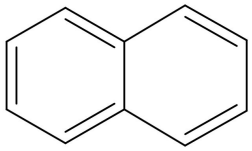
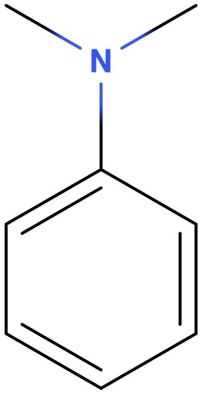
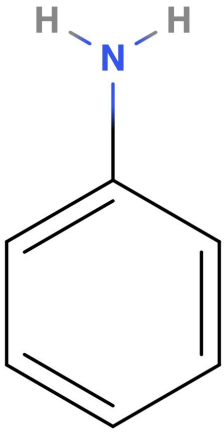


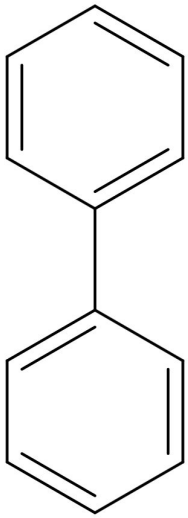
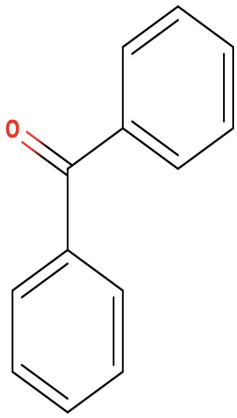
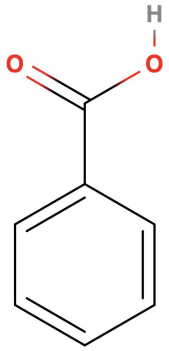
d. Why is it important to use a co-spot?

A co-spot lane is important when performing experiments using TLC plates, because it allows the ability to differentiate between compounds that have similar Rf values. It would be very difficult to identify unknown compounds without a valid co-spot present on the TLC plate. On the co-spot itself, it is imperative that there should be separation between the two molecules in which the R_f values can be taken, to allow them to be compared to that of the unknown molecule.

5. **For each of the following sets of compounds perform the following:.**
   a. **Draw the line structure of each molecule.**
   b. **Arrange them in order of increasing polarity.**
   c. **Explain your reasoning.**
      i. Benzyl alcohol, benzaldehyde, benzyl acetate
      ii. Aniline, N,N-dimethylaniline, Naphthalene
      iii. Benzophenone, Biphenyl, Benzoic acid

Least polar **Most polar**				
i.	Benzaldehyde 	Benzyl acetate 	Benzyl alcohol 	
Reasoning	1. Benzaldehyde is the least polar of all the molecules in this group. Benzaldehyde has aldehyde group at the end of the molecule. Therefore, the molecule does have a level of polarity, but it is determined to be the least of these three. 2. Benzyl acetate is more polar than Benzaldehyde as it has an ester group present, and attached to the molecule. The ester groups provide the molecule with a distinct end. This causes the molecule to have a level of polarity present, however as there is no hydroxyl group it is not the most polar in this group. 3. Benzyl alcohol is the most polar molecule as it has an			

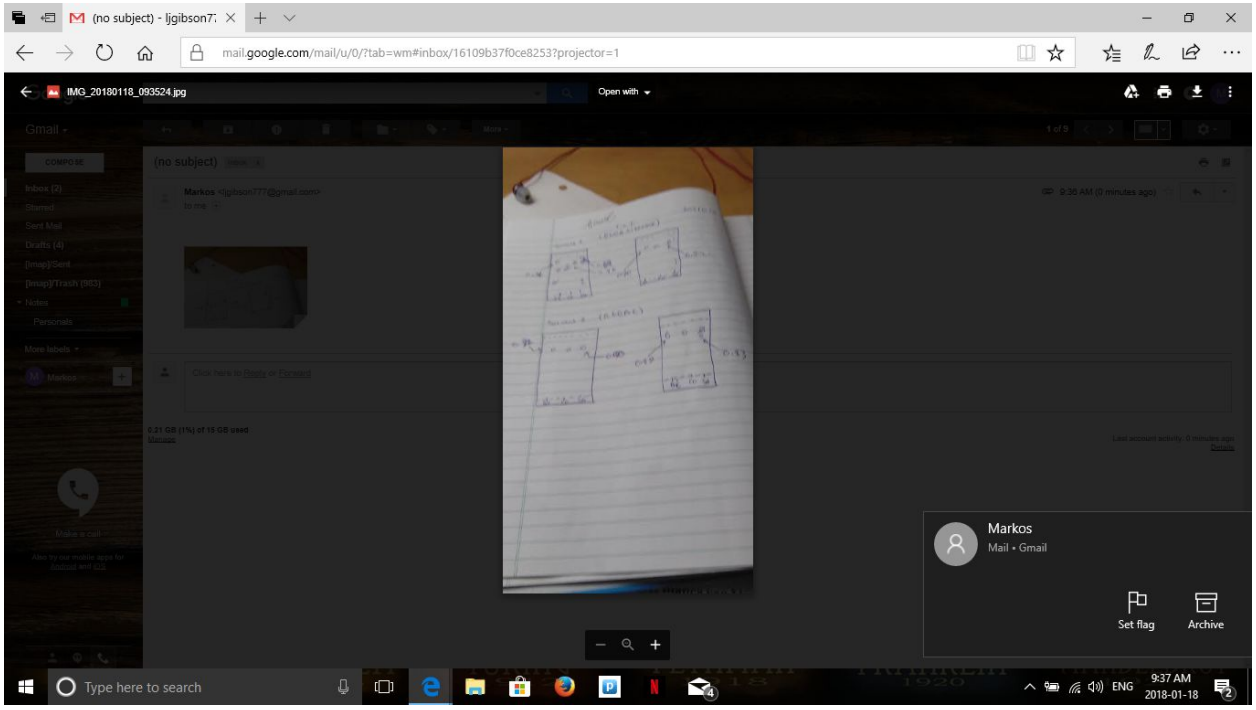
	alcohol functional group. The molecule is small and the OH group creates a distinct end on the molecule, resulting it to be the most polar of all the molecules.			
ii.	Naphthalene 	N,N-dimethylaniline 	Aniline 	
Reasoning	1. Naphthalene is the least polar of this group of molecules. This is because there are no functional groups present. **2.** The N,N-dimethylaniline is more polar than Naphthalene as it has a nitrogen atom present in the molecular structure. However, the nitrogen atom itself is surrounded by carbon atoms, which means that no distinct end is formed in the molecule. Therefore, the polarity level falls short of that of the Aniline molecule. 3. Aniline is the most polar of these three. This is because there is an amino group attached to the phenyl group. The actual Aniline molecule's amino group causes distinct ends on the molecule, resulting in a very polar molecule.			

iii.	Biphenyl 	Benzophenone 	Benzoic acid 	
Reasoning	1. Biphenyl is the least polar molecule out of the three as it has no functional groups and therefore no distinct ends on the molecule to make it polar. 2. Benzophenone has some polarity as a molecule, because it has an oxygen atom. However, it is not an OH group therefore it is not the most polar molecule. 3. Benzoic acid has both an oxygen and hydroxyl group. This creates a very distinct end on the molecule, and causes it to be very polar (more than Biphenyl and Benzophenone).			

Bibliography

Org. Lett. **2018, 20, 24, 7856-7859**

Appendix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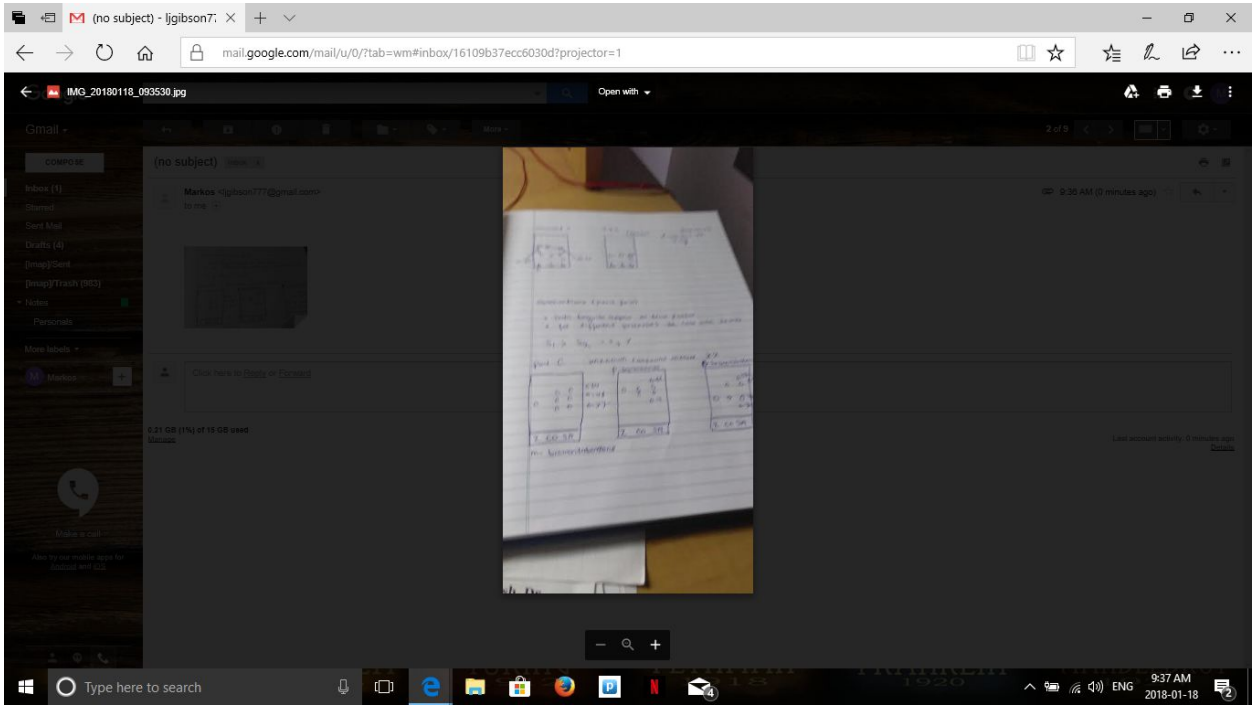
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2018-01-18

Peer Evaluations

COURSE: CHM1321 **TA Name:** Filipe Matos
YOUR NAME (PRINT): LJ Gibson **SIGNATURE:** LJGibson

CONFIDENTIAL PEER EVALUATION FORM FOR EXPERIMENT _____

Each team member must submit one assessment form evaluating each **other** member of the team.
Teams will consist of 2 (max 3) members for reports.

You may edit this form.

Do not share or discuss the contents or possible contents of this assessment with others.

In assessing the work of your fellow team members, consider the following aspects:

- Quality of work
- Contribution to the work as a whole
- Ability to get along with others
- Improvements when asked to correct

Team member name	Comments	Grade (/5)
Mashal Joyaa	- Good new lab mate for semester - Communicated well and on time - Helped out in assisting both of us in learning concepts required for the lab	5

A – Excellent (5) **B: Great (4)** **C: Good (3)** **D: Fair(2)** **F: Poor (1)**

Note: Do not evaluate yourself on this form

COURSE: CHM1321 **TA Name:** Filipe Matos
YOUR NAME (PRINT): Mashal Joyaa **SIGNATURE:** *MashalJoyaa*

CONFIDENTIAL PEER EVALUATION FORM FOR EXPERIMENT _____

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- Quality of work
- Contribution to the work as a whole
- Ability to get along with others
- Improvements when asked to correct

Team member name	Comments	Grade (/5)
LJ Gibson	- Very hard working lab mate! - Helped out a lot in lab execution, and helping me understand what to do - Helped lead the lab team, and made me want to keep doing more to make sure lab got done as well as possible	5

A – Excellent (5) **B: Great (4)** **C: Good (3)** **D: Fair(2)** **F: Poor (1)**

Note: Do not evaluate yourself on this form