Thin layer chromatography

Experiment 1

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Section: Z07

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Materials:

- TLC Plates
- UV light
- Glass capillary
- Developing jar
- Test tubes

Procedure:

The procedure is as in the lab manual (CHM 1321 Organic chemistry lab manual 2019 exp 1) using unknown # 92 in part A and B, and unknown (T) in part C.

Calculations:

Rf: distance the solution travelled / distance the solvent travelled

PART A:

- Plate 1

Reference: 3.00cm/4.60cm =0.65
Co spot: 2.90cm/ 4.60cm =0.63
Sample: 3.00cm/4.60cm =0.65

- Plate 2

Reference: 2.10cm/ 4.60cm =0.45
Co spot: 2.10cm/ 4.60cm =0.45
Sample: 2.20cm/ 4.60cm =0.48

PART B-1:

Plate 1:
Reference: 4.3cm/ 4.7cm = 0.91
Co spot: 4.3cm/ 4.7cm = 0.91
Sample: 4.3cm/ 4.7cm = 0.91

Plate 2:
Reference: 4.5cm/ 4.8cm = 0.94
Co spot: 4.5cm/ 4.8cm = 0.94
Sample: 4.5cm/ 4.8cm = 0.94

PART B-2:

Plate 1:

Reference: 0.9cm/4.7cm =0.19

Co spot: 0.9cm/4.7cm =0.19

Sample: 0.9cm/4.7cm =0.19

Plate 2:

Reference: 0.6cm/4.8cm =0.13

Co spot: 0.6cm/4.8cm =0.13

Sample: 0.4cm/4.8cm =0.08

Observations:

Data tables:

Part A TLC plates with 2:8 mixture of ethyl acetate and hexane

Plate #	Eluent	Reference	Co spot	Sample	R R_f	C R_f	S R_f
1	**Ethyl acetate Hexanes**	**benzophenone**	**Benzophenone Dichloromethane Unknown #92**	**Dichloromethane Unknown #92**	**0.65**	**0.63**	**0.65**
2	**Ethyl acetate Hexanes**	**Biphenyl**	**Biphenyl Dichloromethane Unknown #92**	**Dichloromethane Unknown #92**	**0.45**	**0.45**	**0.48**

Part B-1 : TLC plates with pure Ethyl acetate

Plate #	Eluent	Reference	Co spot	Sample	R R_f	C R_f	S R_f
1	Ethyl acetate	benzophenone	Benzophenone Dichloromethane Unknown #92	Dichloro methane Unknown #92	0.91	0.91	0.91
2	Ethyl acetate	Biphenyl	Biphenyl Dichloromethane Unknown #92	Dichloro methane Unknown #92	0.94	0.94	0.94

Part B-2 : TLC plates with pure Hexanes

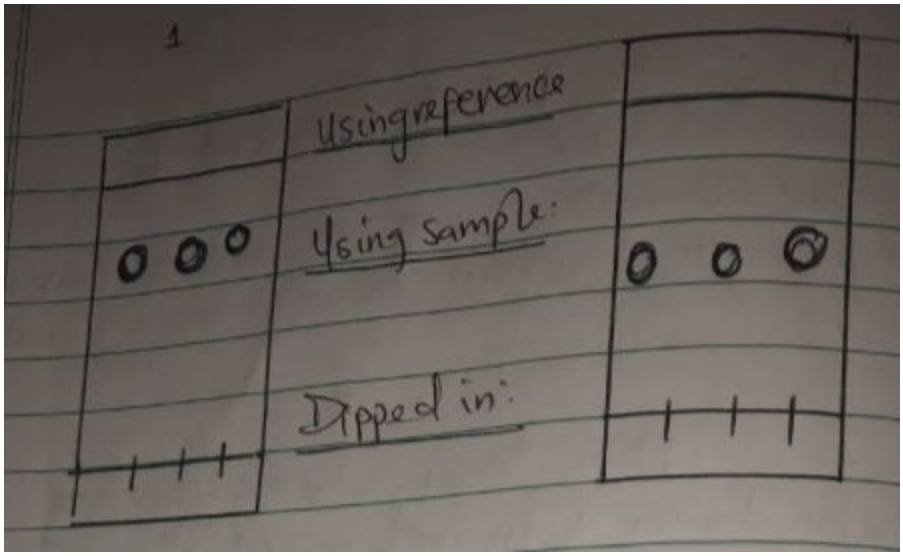
Plate #	Eluent	Reference	Co spot	sample	R R_f	C R_f	S R_f
1	Hexanes	benzophenone	Benzophenone Dichloromethane Unknown #92	Dichloro methane Unknown #92	0.19	0.19	0.19
2	Hexanes	Biphenyl	Biphenyl Dichloromethane Unknown #92	Dichloro methane Unknown #92	0.13	0.13	0.08

Part C : TLC plates with 9:1 Hexanes: Ethyl acetate

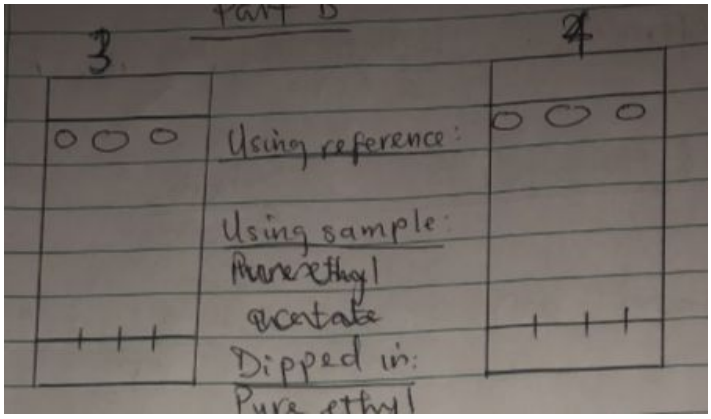
Plate #	Eluent	Reference	Co spot	sample
1	9:1 Hexanes: Ethyl acetate	O	O and T	T
2	9:1 Hexanes: Ethyl acetate	P	P and T	T
3	9:1 Hexanes: Ethyl acetate	M	M and T	T

TLC Plate Figures:

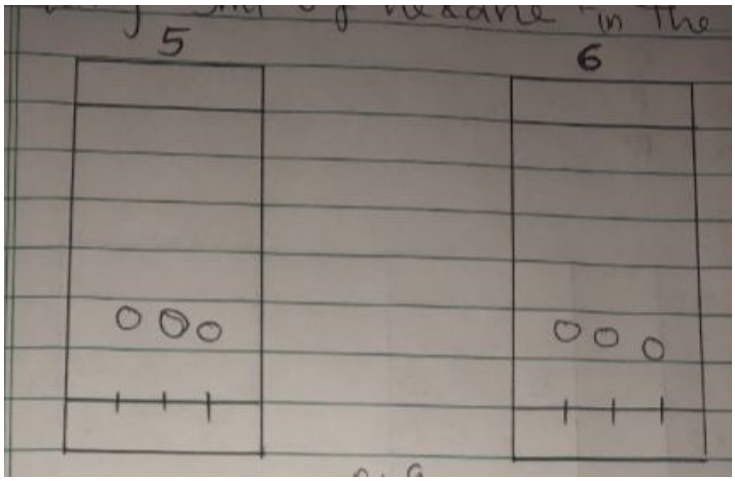
Part A



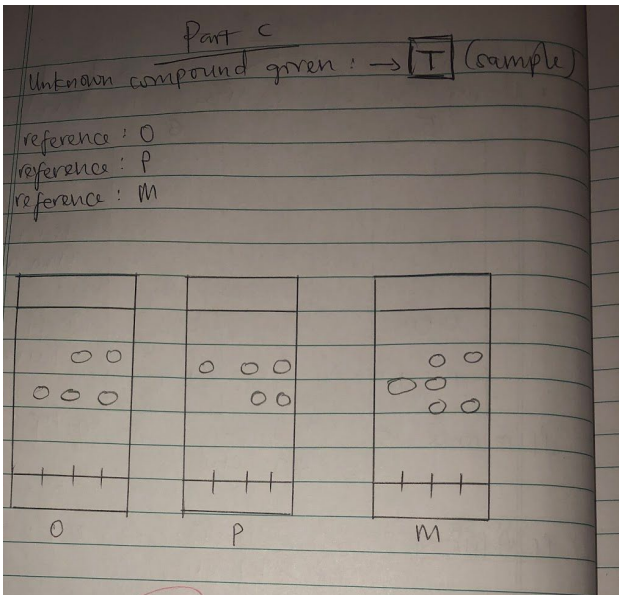
Part B-1



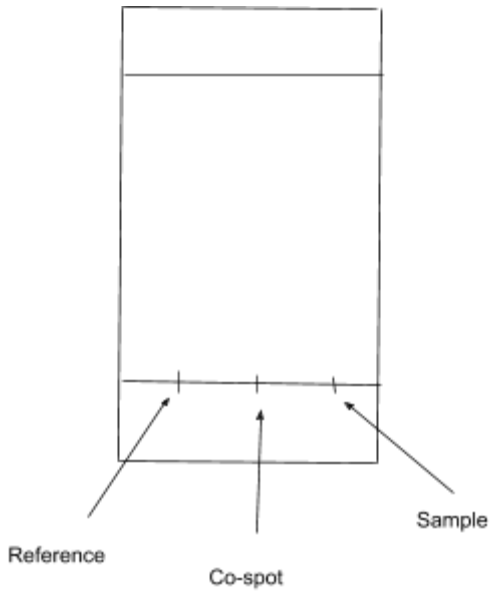
Part B-2



Part C



- All TLC plates abide by the following format:



Discussion:

Part A

- Using unknown #92
- The plates were developed in a 9;1 mixture of ethyl acetate and hexanes
- The Rf values of the unknown sample were very close to the Rf values of both the reference compounds so there is no way of identifying the compounds in the unknown sample.
- It was expected that the unknown sample and the reference compound could be differentiated from one another, but there Rf values were almost identical in both test, and they co-spot did not separate.
- Despite the flaw it is most likely that benzophenone is the unknown sample because the Rf values are relatively close to each other, if the sample was biphenyl the sample spot would have had a Rf differing from the reference and the co spots would have had 2 spots
- Benzophenone should be a more polar compound than biphenyl because benzophenone contains a polar C-O bond, whereas biphenyl contains no polar bonds. This should cause the biphenyl spot to have a higher Rf value than the benzophenone. This is because a less-polar compound will be more attracted to the solvent, since the solvent is less polar than the silica gel on the plate.

Part B

- Using unknown #92

- The plates for part 1 were developed in ethyl benzene.
- The plates for part 2 were developed in hexanes.
- Using pure ethyl acetate the plates had Rf values of 0.91-0.94, and the spots moved far.
- Using pure hexanes the plates had Rf values of 0.08-0.19 and the spots did not move far.
- Ethyl acetate is a more polar solvent than pure hexanes because if its polar C-O bonds and asymmetric structure. Hexanes only have C-H bonds which are not polar. The higher polarity of the ethyl acetate causes the compounds to move up the plate easier, resulting in higher Rf values.

Part C

- We were given unknown mixture T.
- The three plates were developed in a mixture of ethyl acetate and hexane.
- After the TLC plate was developed, there were 2 spots in the unknown sample lane. This means there are at least 2 compounds in that sample.
- The spot for p-bromonitrobenzene had the same Rf as the top spot of the unknown substance. The spot for o-bromonitrobenzene had the same Rf as the bottom spot of the unknown substance. The spot for m-bromonitrobenzene had an Rf between both the unknown spots.
- Unknown substance T is composed of ortho-bromonitrobenzene and para-bromonitrobenzene, as the sample spot has the sample Rf value as those references because they travelled the same distance. The meta-bromonitrobenzene had an Rf value that was not equal to either of the compounds in the sample T.

Sources of Error:

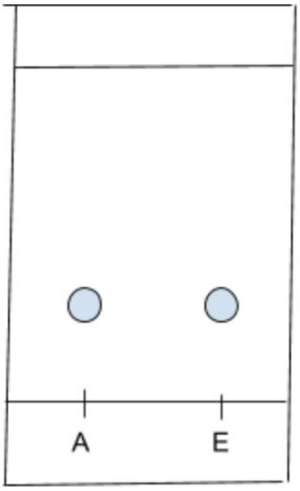
- The centre of the spots are hard to estimate, so the measurements used in the Rf calculations will be slightly inaccurate.
- The solution evaporated quickly and may have caused the the solution line to be drawn inaccurately

Post lab questions:

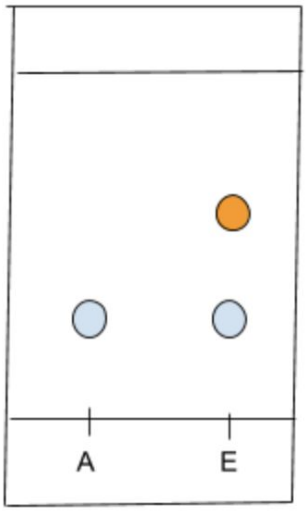
1. It is important to make co-spots last because the co-spot is used for reference, for instance the solvent may run slightly causing identical components to have different heights, in this situation the co spot is used to determine the variation. if they are not done last, the capillaries used will become contaminated with the second substance and both substances will be present in the other lanes.
2. Increasing the polarity of the solvent will increase the Rf values of the TLC test. This is because a polar compound will be attracted to the solvent as well as the silica gel rather than just to the silica. This attraction to the solvent will cause the

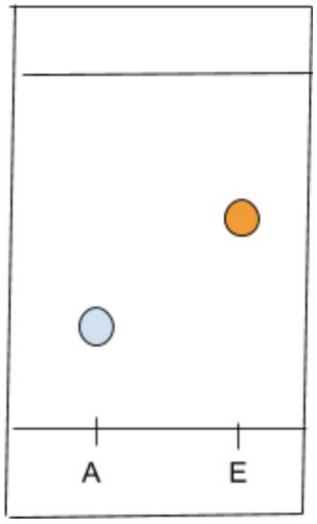
compound to move up the plate with the solvent rather than just being attracted to the silica.

3.

a. 

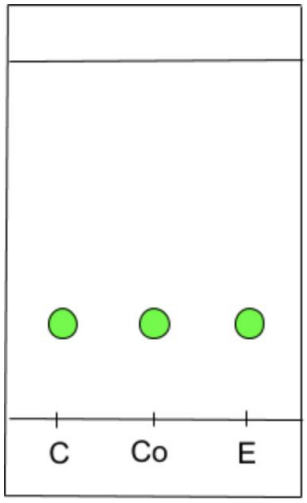
Blue = Compound A
Orange = Compound B
A = Sample A Reference
E = Experimental Sample

b. 

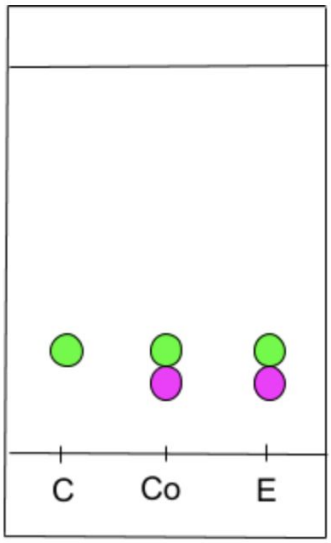
c. 

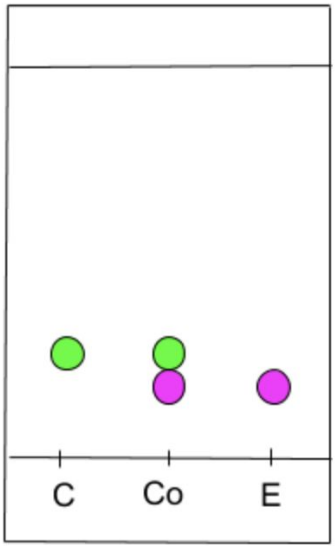
d. It is better to use sample A rather than sample B because the reaction causes A to turn into B and not the other way around. If you started with sample B, there would be no reaction.

4.

a. 

$Green = Sample\ C$
$Pink = Sample\ D$
$C = Sample\ C$
$Co = Cospot\ (C + E)$
$E = Experimental\ Sample$

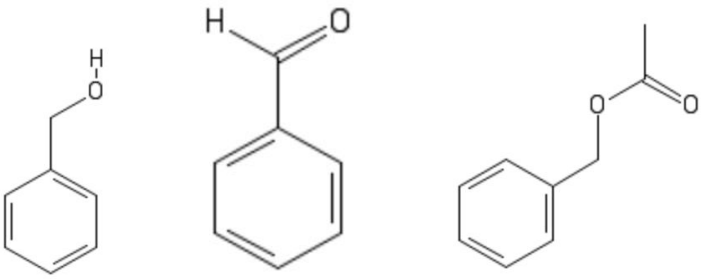
b. 

c. 

d. It is important to use a co-spot because the R_f values are so close (0.40 and 0.43) that it is hard to differentiate between them if they are in different lanes.

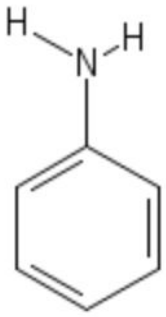
5.

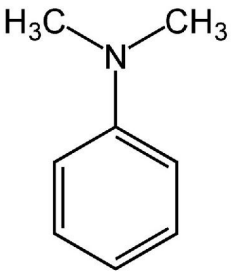
a.

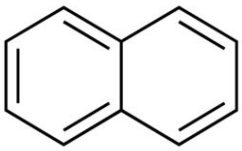
i. 

Benzyl Alcohol Benzaldehyde Benzyl Acetate

ii.

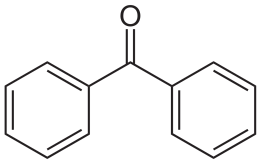


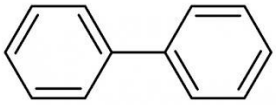


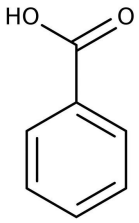


Aniline N, N-dimethylaniline Naphthalene

iii.







Benzophenone Biphenyl Benzoic Acid

b.

i. Benzyl Acetate, Benzyl Alcohol, Benzaldehyde
ii. Aniline, N, N-dimethylaniline, Naphthalene
iii. Benzoic Acid, Benzophenone, Biphenyl

c.

i. Benzyl acetate is the most polar because it contains 2 C-O bonds near each other. A C-O bond has a Δ Electronegativity of 1. This is less that the O-H bond in benzyl alcohol, but there are 2 C-O bonds in benzyl acetate so it is likely more polar. The second most polar is benzyl alcohol because of its O-H bond with a ΔEN of 1.4. The third most polar is benzaldehyde because it only has one C-O bond with a Δ EN of 1.
ii. Aniline is the most polar because it contains 2 N-H bonds with a ΔEN of 1.9. N, N-dimethylaniline and naphthalene are both nonpolar because they have no polar bonds.
iii. Benzoic acid is the most polar because is has an O-H bond with a ΔEN of 1.4 as well as a C-O bond with a ΔEN of 1. Both of these polar bonds are on the same side. The second most polar is benzophenone because it has a C-O bond with a ΔEN of 1. Biphenyl is the least polar because it has no polar bonds.

Conclusion:

Part A

- The unknown compound #92 could not be identified because its Rf values were too close to the reference compounds to be differentiated.

Part B

- Using ethyl acetate as a solvent, the Rf values of the compounds ranged between 0.91-0.94.
- Using hexanes as a solvent, the Rf values of the compounds ranged between 0.08-0.13.
- Ethyl acetate was found to be more polar than hexanes.

Part C

- The unknown compound T was identified as ortho bromonitrobenzene and meta bromonitrobenzene.

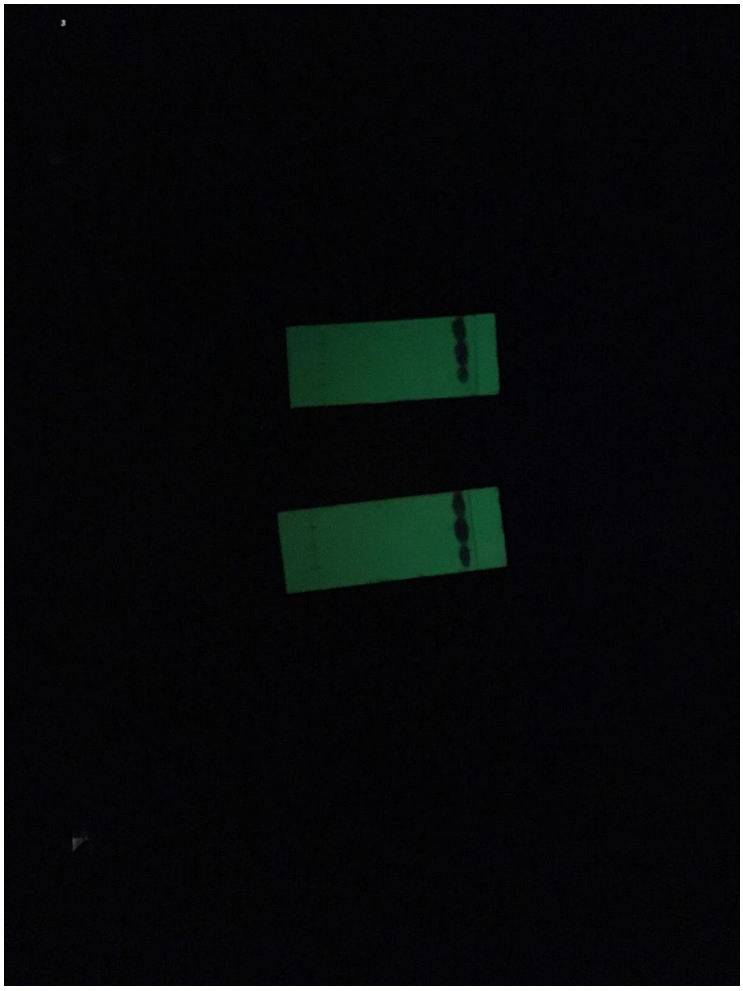
References:

- Images for Post-Lab Question 5 were made using PubChem Sketcher V2.4
- "CHEM 344 Thin Layer Chromatography." *WISC*, University of Wisconsin-Madison.

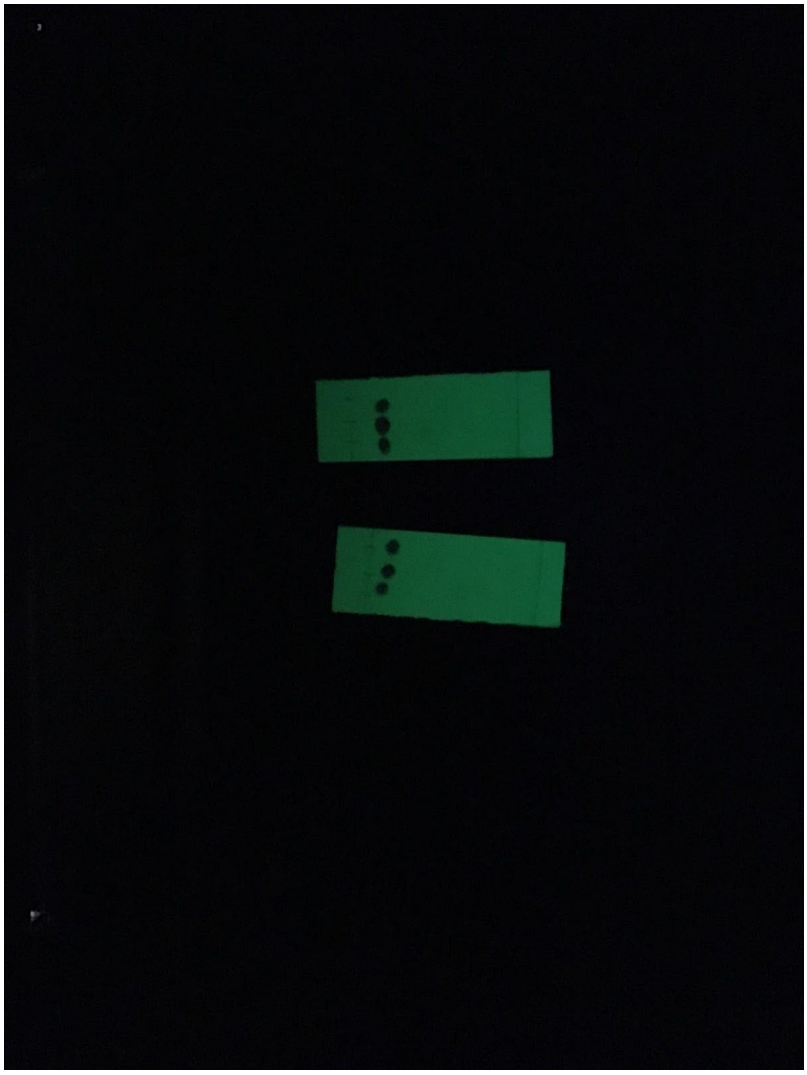
Figures from UV light

Part A:

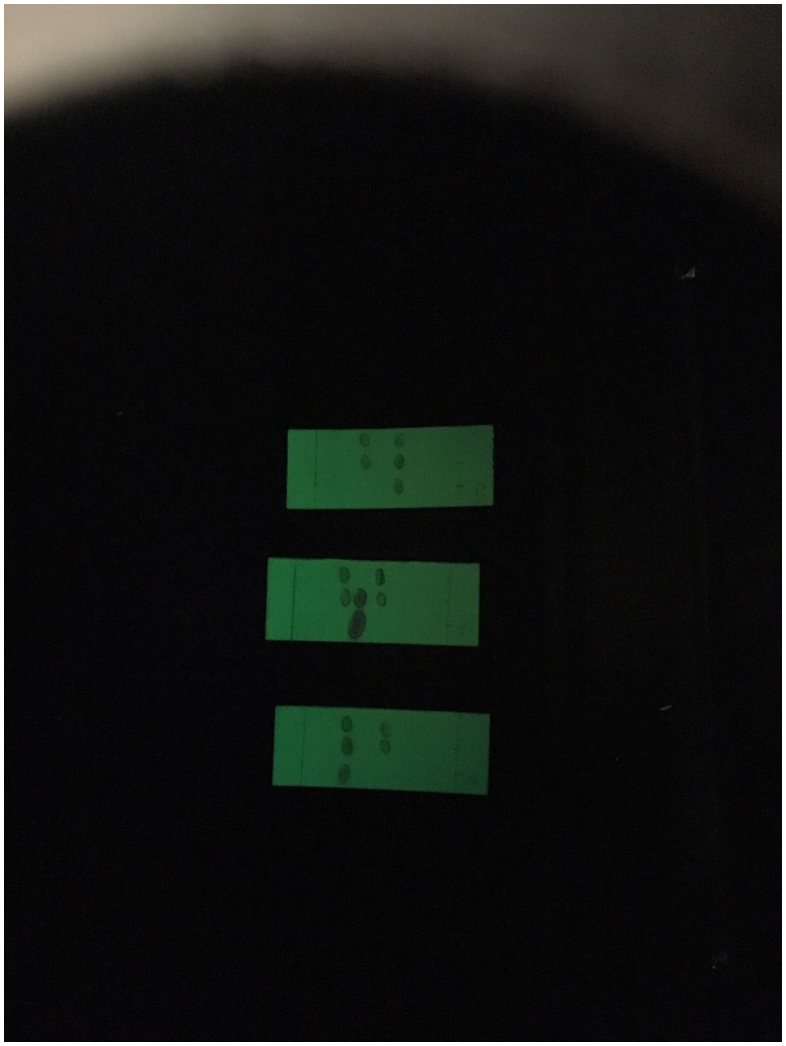
Part B-1:



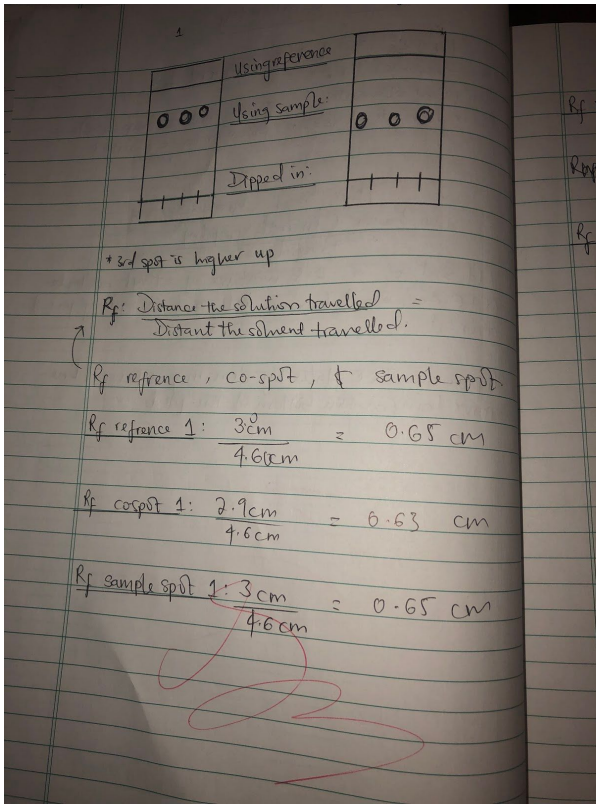
Part B-2:



Part C:


Ortho-bromonitrobenzene
Meta-bromonitrobenzene
Para-bromonitrobenzene

Raw Data:



* 3rd spot is higher up

Rf: Distance the solution travelled / Distant the solvent travelled.

Rf refrence, co-spot, & sample spot

Rf refrence 1: 3cm / 4.6cm = 0.65 cm

Rf cospot 1: 2.9cm / 4.6cm = 0.63 cm

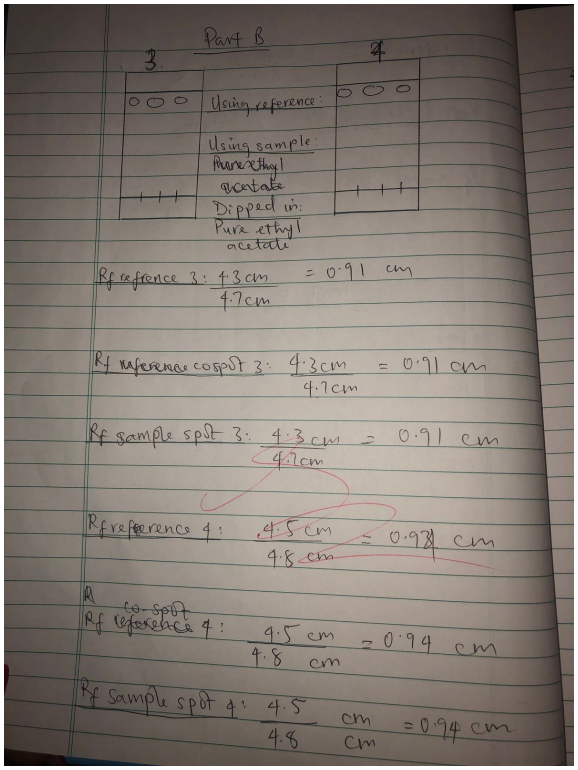
Rf sample spot 1: 3 cm / 4.6 cm = 0.65 cm

Rf refrence 2: 2.1cm / 4.6cm = 0.45 cm

Rf refrence Rf co-spot 2: 2.1cm / 4.6cm = 0.45 cm

Rf sample spot 2: 2.2cm / 4.6cm = 0.48 cm

Part B



Rf refrence 3: 4.3cm / 4.7cm = 0.91 cm

Rf refrence cospot 3: 4.3cm / 4.7cm = 0.91 cm

Rf sample spot 3: 4.3 cm / 4.7cm = 0.91 cm

Rf reference 4: 4.5 cm / 4.8 cm = 0.94 cm

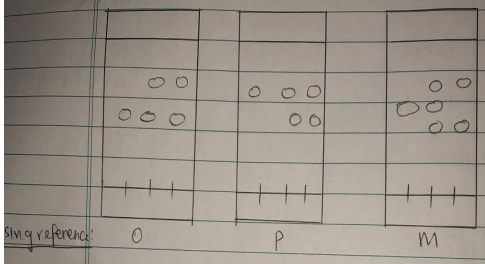
Rf reference 4: 4.5 cm / 4.8 cm = 0.94 cm

Rf Sample spot 4: 4.5 cm / 4.8 cm = 0.94 cm

Part C

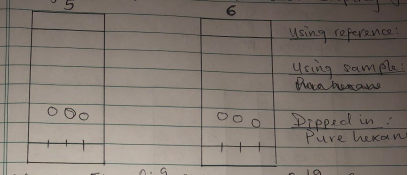
Unknown compound given: → T (sample)

reference: O
reference: P
reference: M



Part B.2

* ~~Unknown compound given~~ → I
* Using 5ml of hexane in the developing



Rf reference 5: $\frac{0.9\ cm}{4.7\ cm} = 0.19\ cm$

Rf ~~reference~~ co-spot 5: $\frac{0.9\ cm}{4.7\ cm} = 0.19\ cm$

Rf sample spot 5: $\frac{0.9\ cm}{4.7\ cm} = 0.19\ cm$

Rf reference 6: $\frac{0.6\ cm}{4.8\ cm} = 0.13\ cm$

Rf co-spot 6: $\frac{0.6\ cm}{4.8\ cm} = 0.13\ cm$

Rf sample spot 6: $\frac{0.4\ cm}{4.8\ cm} = 0.08\ cm$