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JANUARY 18, 2016

CHM1321 A1

LAB REPORT

EXPERIMENT #1

THIN LAYER CHROMATOGRAPHY

UNIVERSITY OF OTTAWA

Introduction:

In this experiment, we used a technique called thin layer chromatography. This technique is used to identify the compounds in a mixture by separating the mixture based on solubility and polarity. It is also used to monitor the progression of a reaction, and when a reaction comes to an end. Levels of purity can be changed according to the experimenter's want by changing the solvent (eluent). However, a mixture of polar and non-polar solvents in variable amounts can also be used. These kinds of mixtures will give us the accurate polarity. Also, the polarity of the compound to be purified should be considered when performing a TLC experiment. In part A of the experiment, we used TLC to identify the components of an unknown mixture (#35). In part B, we examined the effect of different solvents on TLC. Finally in part C, we used TLC to determine the ratio of compounds in the mixture ZZ.

Procedure:

The same procedure in the lab manual has been used. Refer to lab manual pages (16-18). However for part C, a 9:1 mixture of hexane and ethyl acetate was used as the solvent system.

Observations:

- Unknown mixture (#35) dissolved quickly in dichloromethane, giving a colorless solution with a very faint sweet odor resulting from the dichloromethane.
- The mobile solvent (mixture of ethyl acetate and hexane 2:8) elutes fairly fast across both plates (the one with benzophenone as reference solution, and the one with biphenyl) in part A.
- For part B, when 2 plates with reference points (benzophenone and biphenyl) were set in ethyl acetate, the elution period was longer than that of part A.
- In the second part of section B, when another 2 plates were developed in a hexane solvent, the plate with biphenyl as a reference point did not elute as much as the one with benzophenone; the

mobile phase goes about half the distance of that of the benzophenone.

- For part C, the mobile phase elutes at almost the same speed in all four TLCs that have different reference points.

Discussion:

This experiment consists of three parts, in which we use the thin layer chromatography (TLC) technique and calculated retention factors in each of the parts for a different purpose. The retention factor is the distance travelled by a compound divided by the distance travelled by solvent front. The larger the retention factor of a compound, the greater distance it travels on the plate. In addition, higher retention factors (R_f) indicate that the compound is less polar, which means it reacts less vigorously with the silica in the plate. Thus it moves faster through the silica gel. While lower retention factors mean that the compound is highly polar and does not move great distances towards the solvent front.

$$R_f = \frac{Distance\ travelled\ by\ compund}{Distance\ travelled\ by\ solvent\ front}$$

In part A of the experiment, we use TLC to identify the components of an unknown mixture (#35). We prepared two TLC plates, each one with a different reference solution spot being benzophenone and biphenyl, but the same sample mixture dissolved in dichloromethane. For the TLC plate with benzophenone as the reference solution, the sample spot and the reference solution have almost the same retention factor, with the reference having an R_f of 0.45 and the sample spot 0.48. This shows that the mixture contains the compound benzophenone. For the plate with biphenyl as the reference solution, the sample spot and the reference spot also travel about the same distance. The R_f of biphenyl is 0.13 and that of the sample is 0.12. This proves that the mixture contains biphenyl in addition to benzophenone. Also, other spots at different levels appear in the co-spot lane. This shows that the sample contains more than just biphenyl and

benzophenone. Thus, the mixture has impurities. The TLC plate with benzophenone appears more like an upside-down water drop, because the apparatus may have been disturbed; it may have been accidently touched. From the high R_f of benzophenone we can conclude that it is less polar than biphenyl. However, biphenyl, which has a lower R_f is considered highly polar because it slightly moves towards the solvent front.

In part B of the experiment, we determine the effect of different solvents (eluants), which are ethyl acetate (EtOAc) and hexane, on TLC plates with the same sample and reference solutions as those in part A. The TLC plates that were put in the jar with the highly polar solvent, ethyl acetate, show that the mixture contained both benzophenone and biphenyl, since the reference spots and sample spots are on almost the same level on the TLC plates. However on the TLC plate with benzophenone, the reference solution is only slightly higher than the sample spot, causing a difference of 0.08 in the retention factor.

When two TLC plates, with benzophenone and biphenyl as reference spots and the same sample solution of the unknown mixture (#35) dissolved in dichloromethane as sample spot, were put in an eluant of hexanes, which is less polar than ethyl acetate, the reference points appeared to stay in the same place (on the origin line). However, this is unusual since hexanes are considered to have very low polarity, and solvents with low polarity travel higher and faster through the TLC plate. On the other hand, if a sample mixture was put in a highly polar solvent, the solvent will spend most of the time reacting with the sample mixture. Therefore it will not have a high solvent front, and most substances would have equal solubility or insolubility, and therefore the spots would be on almost the same level. The co-spots and sample spots for the plates in hexanes, appeared to move upward in both plates, and the co-spot and sample spot of each TLC plate where on the same level. In the TLC plate with the reference solution biphenyl, the solvent front stopped rising upward after moving half-way through the TLC plate.

According to the previous paragraph, I conclude that we have mixed up the labeling of solvents for the two sections of part B, which resulted in opposite

results. Therefore the first part should be the plates in hexane, and the second, the plates that are in ethyl acetate.

For part C of the experiment, the TLCs show that only m-bromonitrobenzene is in the mixture, since it is the only one that has a reference and sample spot on the same level. For p-bromonitrobenzene, the solvent front was slanted probably due to a disturbance when the TLC plate was in the jar. The reference spot also appears to be smeared. This may be because the reference lane was overloaded. For the TLC with bromobenzene as a reference solution, no reference spot appeared above the origin line. This might be due to the decomposition of the reference compound on the silica. In conclusion the only mixture that seems to be in the compound ZZ is m-bromonitrobenzene, along with other impurities.

To find %moles of m-bromonitrobenzene:

Substitute %peak 3(SEE CALCULATIONS IN CALCULATIONS' SECTION) in

y=0.976x +0.804

Y=0.976(57.7%) + 0.804

Y= 1.36% moles of m-bromonitrobenzene

The calculated % Area of peak 3 (m-bromonitrobenzene) 57.7%, is represented by the calibration curve "Amount of the *meta-para* mixture of bromonitrobenzene". After substituting the percentage area in the equation, the percentage of moles of m-bromonitrobenzene in the compound (ZZ) is 1.36%.

Part A			
Eluent: 2:8 Ethyl acetate and hexanes	**Ref. Solution: Biphenyl**	**Spot**	**R_f**
		Reference spot	0.13
		2^{nd} spot from left	0.43
		3^{rd} (upper) spot	0.37
		4^{th} (bottom) spot	0.25
		5^{th} spot (sample)	0.12
	Ref. solution: Benzophenone	Reference spot	0.45
		Co-spot	0.48
		Sample spot	0.48

Part B							
Eluant: Ethyl acetate (EtOAc)	**Ref. solution: Benzophenone**	**Spot**	**R_f**	**Eluant: Hexane**	**Ref. solution: Benzophenone**	**Spot**	**R_f**
		Ref. spot	0.86			Ref. spot	0*
		Co-spot	0.86			Bottom co-spot	0
		Sample spot	0.78			Upper co-spot	0.5
	Ref. Solution: Biphenyl	Ref. spot	0.86			Bottom sample spot	0
						Upper sample spot	0.46
		2^{nd} spot from left	0.84		**Ref. Solution: Biphenyl**	Ref. spot	0
		3^{rd} spot from left	0.82			Bottom co-spot	0
		4^{th} spot from left(sample spot)	0.84			Upper co-spot	0.62
						Bottom sample spot	0
						Upper sample spot	0.4

*spot remains on the bottom line; did not elute by solvent

Part C			
Eluant: 9:1 hexane acetate	**Ref. Compound: Bromobenzene**	**Spot**	**R_f**
		Ref. spot	Does not appear
		Lower spot	0.25
		Upper co-spot	0.34
		Upper sample spot	0.36
	Ref. Compound: o-bromonitrobenzene	Ref. spot	0.67
		Lowest co-spot	0.27
		Middle co-spot	0.53
		Top co-spot	0.64
		Lowest sample spot	0.27
		Highest sample spot	0.55
	Ref. Compound: p-bromonitrobenzene	Ref. spot	0.75
		2^{nd} point from left	0.15
		3^{rd} point from left	0.15
		Sample point	0.15
	Ref. Compound: m-bromonitrobenzene	Ref. spot	0.5
		Lower co-spot	0.35
		Upper co-spot	0.5
		Lower sample spot	0.38
		Upper sample spot	0.5

Calculations:

How I calculated R_f values:

$$R_f = \frac{Distance\ travelled\ by\ compund}{Distance\ travelled\ by\ solvent\ front}$$

For m-bromonitrobenzene:

Lane 1

%peak 1= [area of peak 1 /(area of peak 1+ area of peak 2)]*100

%peak 2= [area of peak 2 /(area of peak 1+ area of peak 2)]*100

Lane 2

%peak 3 = [area of peak 3/(area of peak 3+ area of peak 4)]*100

%peak 4= [area of peak 4 /(area of peak 3+ area of peak 4)]*100

Area of peak 1 = 5966.309

Area of peak 2 = 4232.652

Area of peak 3 = 5166.208

Area of peak 4 = 3786.359

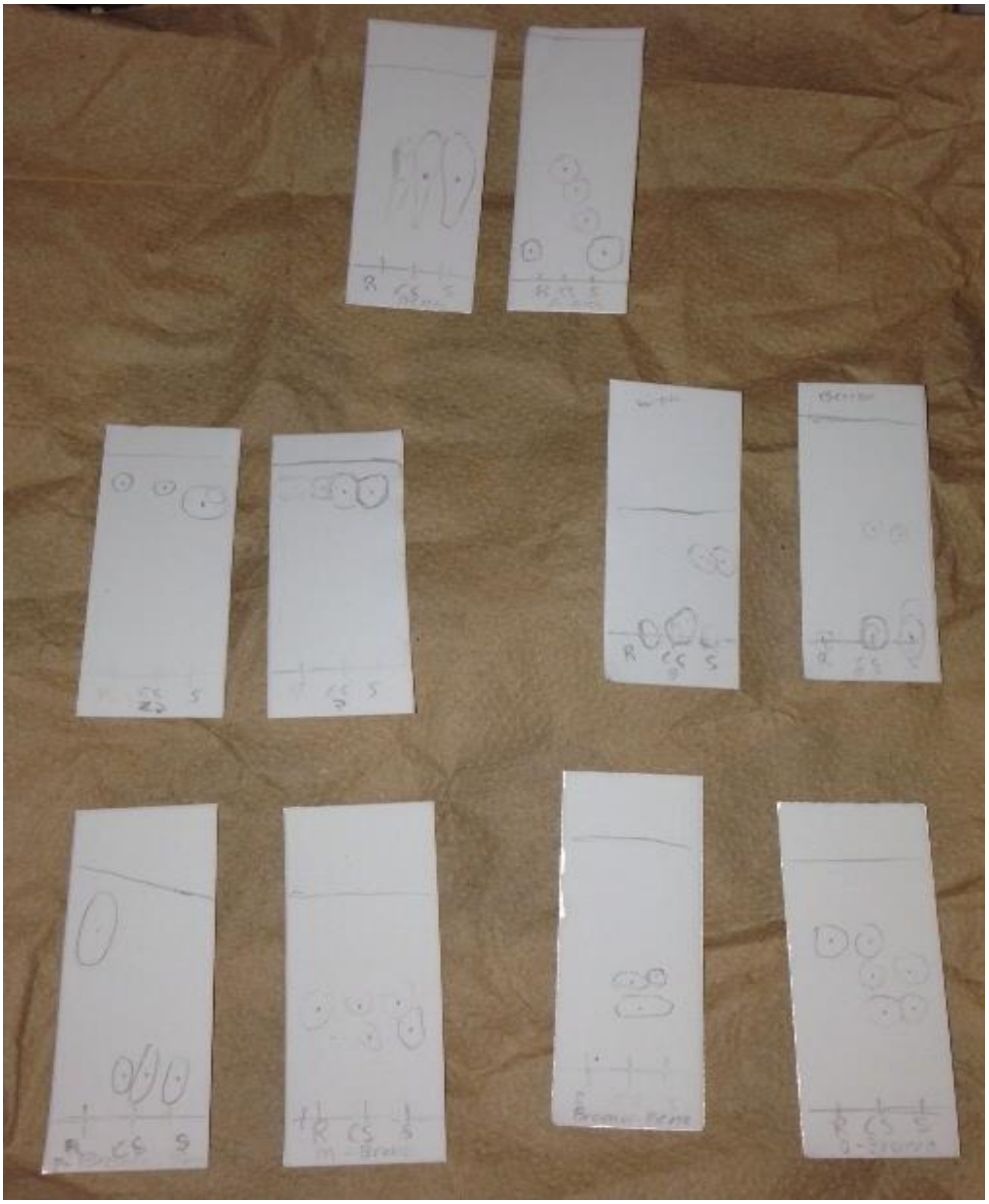
%peak 1 = 58.5%

%peak 2 = 41.5%

%peak 3 = 57.7%

%peak 4 = 42.3%

TLC Plates:



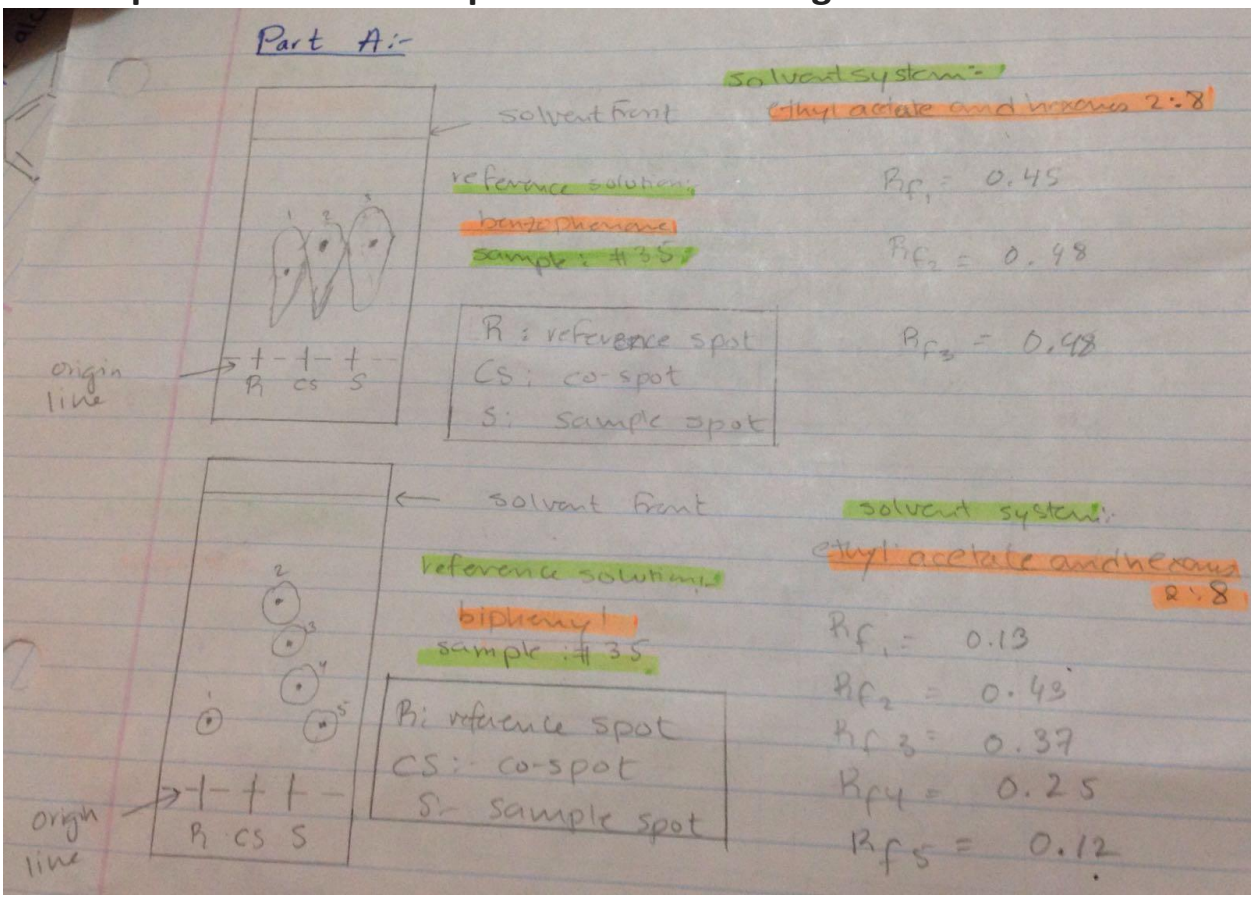
1st row:part A
TLC plate with reference Benzophenone on left, and biphenyl on right

2nd row: part B
Left part: TLC plates in ethyl acetate solvent
with the one on the left being benzophenone and on the right biphenyl

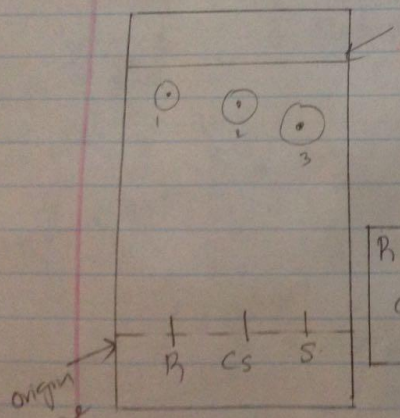
Right part: TLC plates in hexane solvent with biphenyl on left and benzophenone on right

3rd row: part C
From left to right:
P-bromonitrobenzene, m-bromonitrobenzene, bromobenzene, o-bromonitrobenzene

All TLC plates drawn from picture with labeling:



Part B:-



reference solution:- benzophenone

sample: #35

R: reference spot
CS: co-spot
S: sample spot

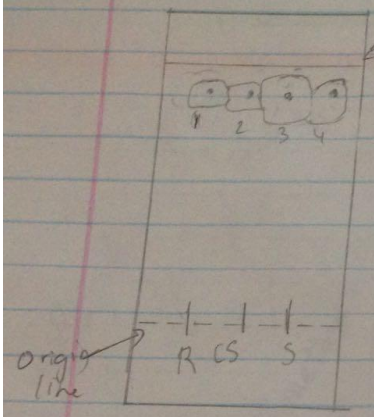
solvent system:- ethyle acetate

$R_{f_1} = 0.86$

$R_{f_2} = 0.86$

$R_{f_3} = 0.78$

Hilroy



reference solution:- biphenyl

sample #35

R:- reference spot
CS:- co-spot
S:- sample spot

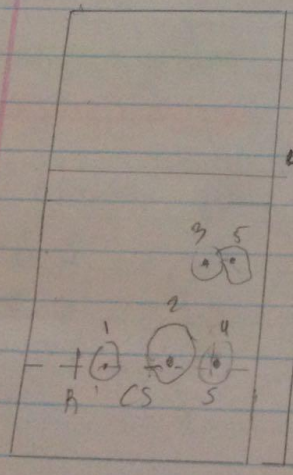
solvent system:- ethyle acetate

$R_{f_1} = 0.86$

$R_{f_2} = 0.84$

$R_{f_3} = 0.82$

$R_{f_4} = 0.84$



reference solution:- biphenyl

sample # 35

R:- reference spot
CS:- co-spot
S:- sample spot

solvent system:- hexane

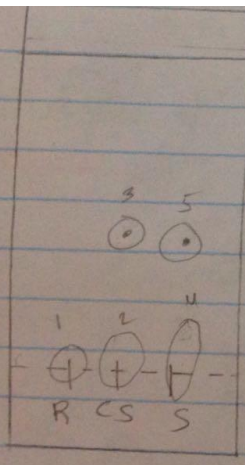
$R_{f_1} = 0$

$R_{f_2} = 0$

$R_{f_3} = 0.62$

$R_{f_4} = 0$

$R_{f_5} = 0.4$



← solvent front

reference solution:-
benzophenone

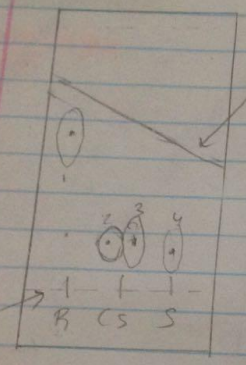
sample #35

R:- reference spot
CS:- cospot
S:- sample spot

solvent system:-
hexane.

$R_{f_1} = 0$
$R_{f_2} = 0$
$R_{f_3} = 0.5$
$R_{f_4} = 0$
$R_{f_5} = 0.46.$

Part C:-



solvent front.

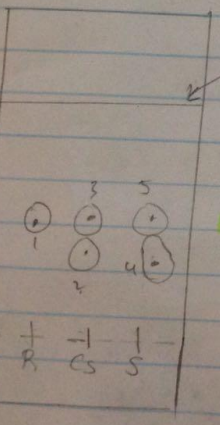
reference solution:-
P- bromonitrobenzene

sample: 22

R:- reference spot
CS:- co-spot
S:- sample spot

solvent system:-
9:1 hexane acetate mixture

$R_{f_1} = 0.75$
$R_{f_2} = 0.15$
$R_{f_3} = 0.15$
$R_{f_4} = 0.15$



solvent front

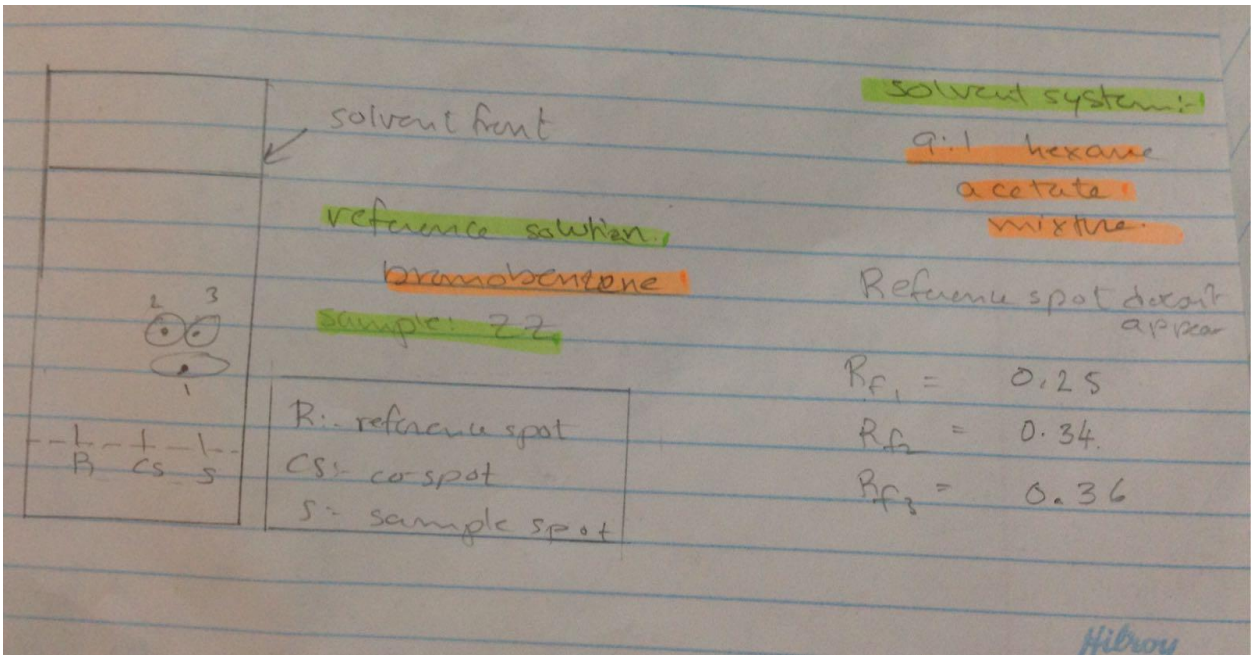
reference solution:-
m- bromonitrobenzene

sample: 22

R:- reference spot
CS:- co-spot
S:- sample spot

solvent system:-
9:1 hexane acetate mixture

$R_{f_1} = 0.5$
$R_{f_2} = 0.35$
$R_{f_3} = 0.5$
$R_{f_4} = 0.38$
$R_{f_5} = 0.5$



solvent system:-
9:1 hexane acetate mixture.

reference solution:
bromobenzene

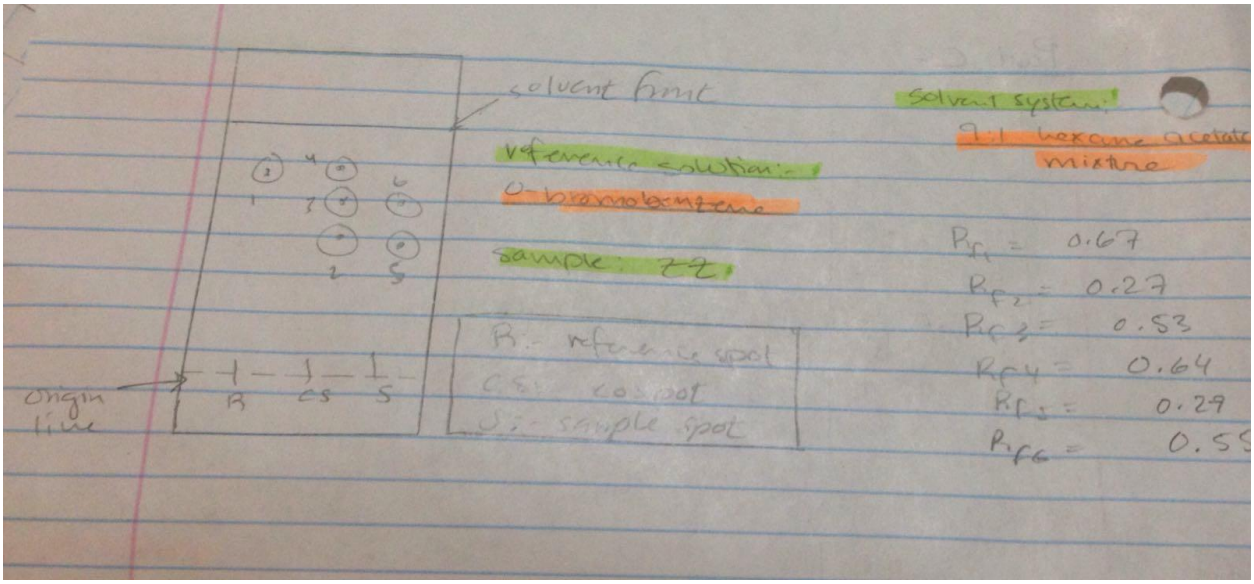
sample: Z2

Reference spot doesn't appear

R_{f_1} = 0.25

R_{f_2} = 0.34

R_{f_3} = 0.36



solvent system:
9:1 hexane acetate mixture

reference solution:
o-bromobenzene

sample: Z2

R_{f_1} = 0.67

R_{f_2} = 0.27

R_{f_3} = 0.53

R_{f_4} = 0.64

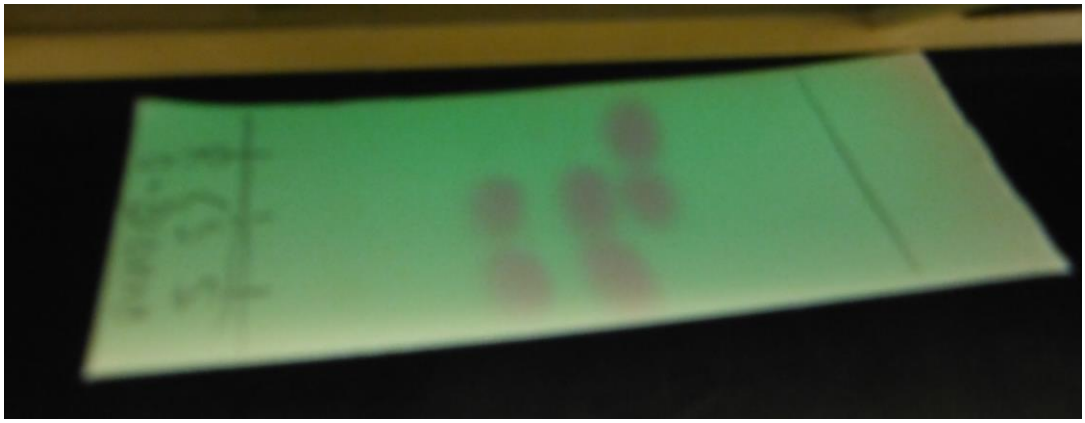
R_{f_5} = 0.29

R_{f_6} = 0.55

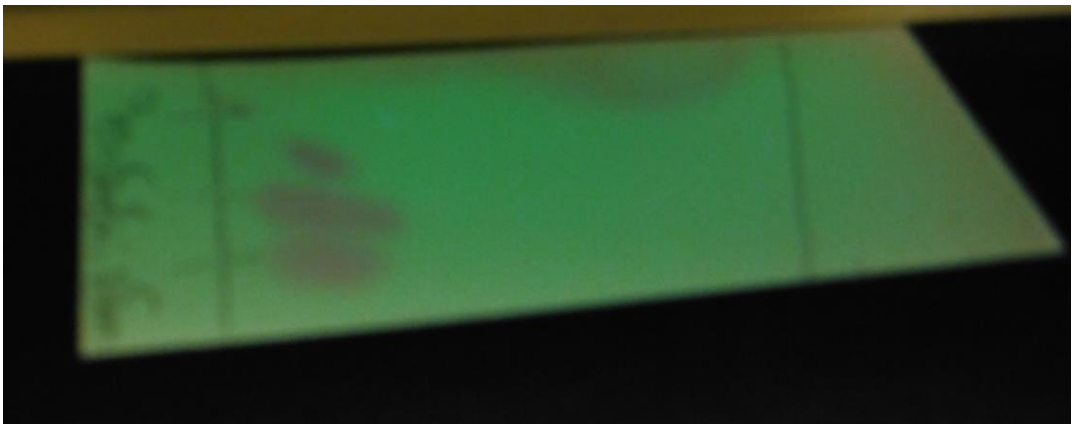
Part C TLC images under UV

Bromobenzene

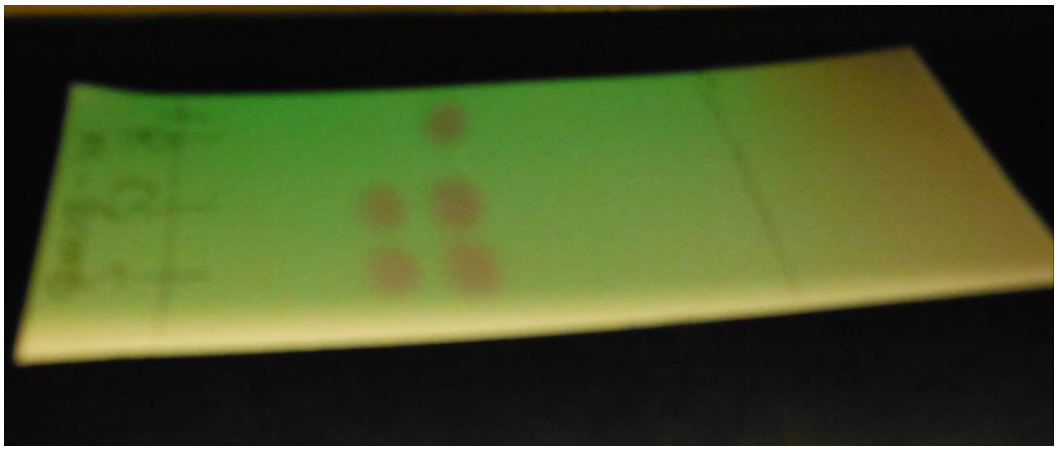
O-bromonitrobenzene



p-bromonitrobenzene



m-bromonitrobenzene



Questions:

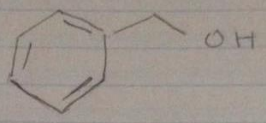
1. When the polarity of a solvent system increases, it becomes more powerful. Thus, it's eluting speed decreases. For example, if we had polar molecules, they will react with silica, therefore staying in their place on the origin line or moving slightly upward. The solvent front will not move a great distance.

2. a: Benzyl alcohol will have the smallest R_f value because it is the most polar compound, and its molecules will interact with silica sticking to it and staying on the origin line or barely move upward.

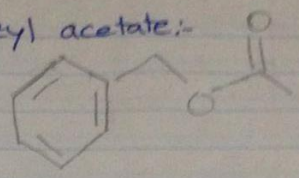
   b: Aniline will have the smallest R_f because it has the highest polarity of all three compounds.

   c: Benzoic acid would have the lowest R_f on silica gel because it is an acid, and has a higher polarity than benzophenone which is a ketone, and biphenyl which has no functional group.

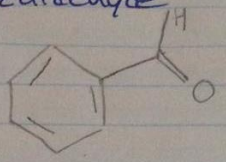
2 a:- benzyl alcohol



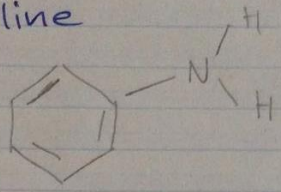
benzyl acetate:-



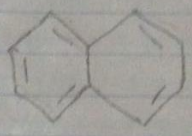
benzaldehyde



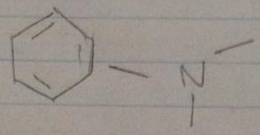
b:- aniline



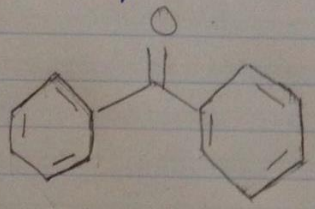
naphthaline



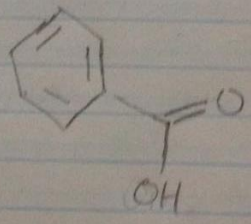
N, N - dimethylaniline



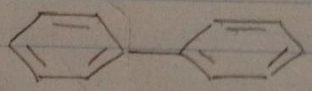
c:- benzophenone



benzoic acid



biphenyl



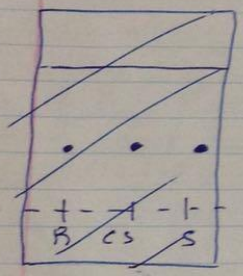
Experiment 1:- Unknown: N= 35

Part A — 10mL Ethylacetate

ethyle acetate used is 9:1 mixing solvent

Unknown cpd:- 1.5

Benzophenone



The 3 spots are almost on the same level, this ~~can~~ shows that it can be the same cpd

R_f for R ≈ 2 cm.

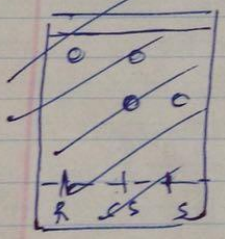
R_f for CS ≈ 2 cm

R_f for S ≈ 2 cm

(Eluted slower than biphenyl).

for GC.

Biphenyl

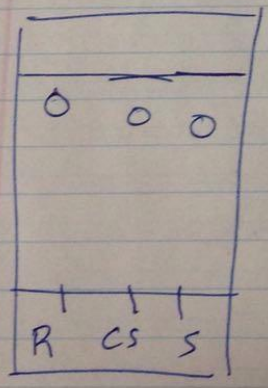


12/1/16 R_f for R ≈ 3.8 cm

R_f for CS ≈ 2.2 cm ≈ 3.8 cm.

R_f for S ≈ 2.3 cm

Part B :- Benzophenone.



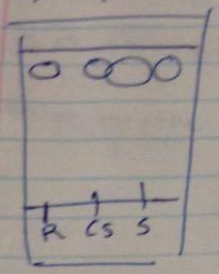
R_f R ≈

R_f CS ≈

R_f S ≈

Hilroy

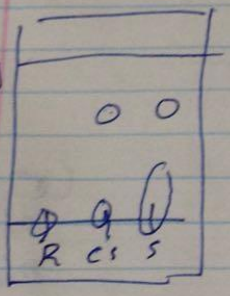
Biphenyl



R_f R =
R_f Cs =
R_f S =

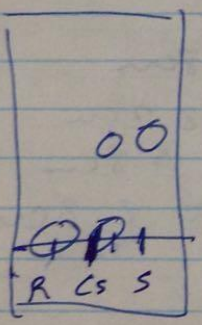
Hexane

~~biphenyl~~ benzophenone



R_f R =
R_f Cs =
R_f S =

~~benzophenone~~ biphenyl



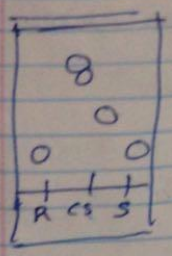
R_f R =
R_f Cs =
R_f S =

Part C = 22

12/1/16

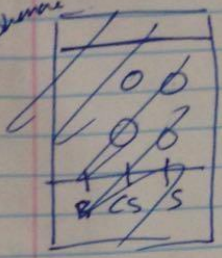
Part A of right mixture.

biphenyl

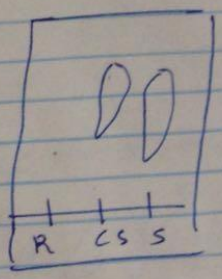


R_f R =
R_f S =
R_f • CS =

benzophenone



R_f R =
R_f S =
R_f CS =



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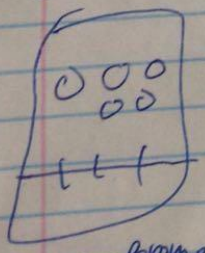
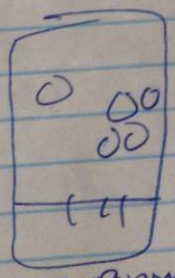
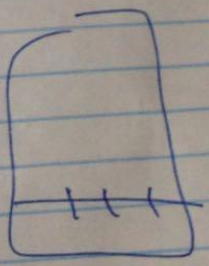
Part C

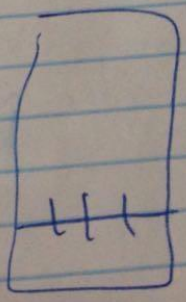


m-Bromo



o-Bromo





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