LAB 1

Thin Layer Chromatography

Name

Lab Partner : _____

CHM 1321 Section A05

Demonstrator: _____

January 10, 2018

Department of Chemistry

University of Ottawa

PROCEDURE AND OBSERVATION :

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

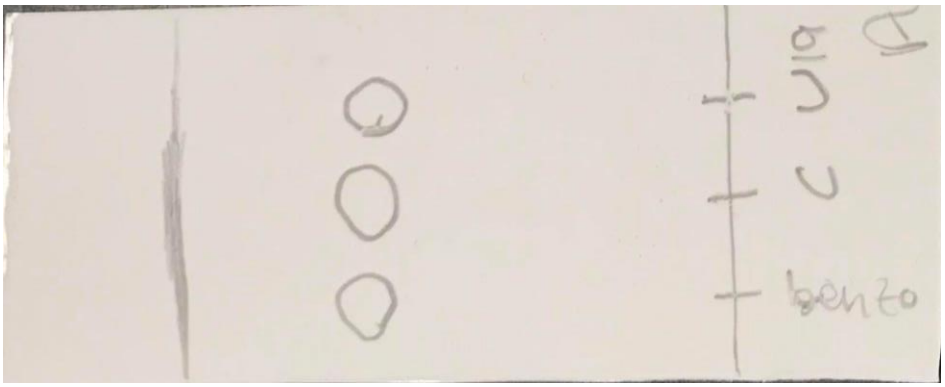
Table of reagents:

Compound	*Molecular Weight (g/mol)*	*Amount*
Ethyl acetate	88.1	2 mL
Hexane	86.0	8 mL
Benzophenone	182.2	2 mL
Biphenyl	154.2	2 mL
Dichloromethane	84.9	2 mL
Unknown 19	unknown	10 mg

Procedure is as described in lab manual (*Experiment 1: Thin Layer Chromatography*. Department of Chemistry. (September 2014). Ottawa: University of Ottawa.)

- Make the 10 mL of 2:8 mixture of ethyl acetate and hexane by adding 2 mL of ethyl acetate and 8 mL of hexane in developing jar
- Use 2 mL of dichloromethane to dissolve 10 mg of unknown 19
- Make sure to spot the benzophenone/biphenyl on the left spot before the co- spot (so the capillary tube doesn't get tainted by the unknown on the co-spot position)

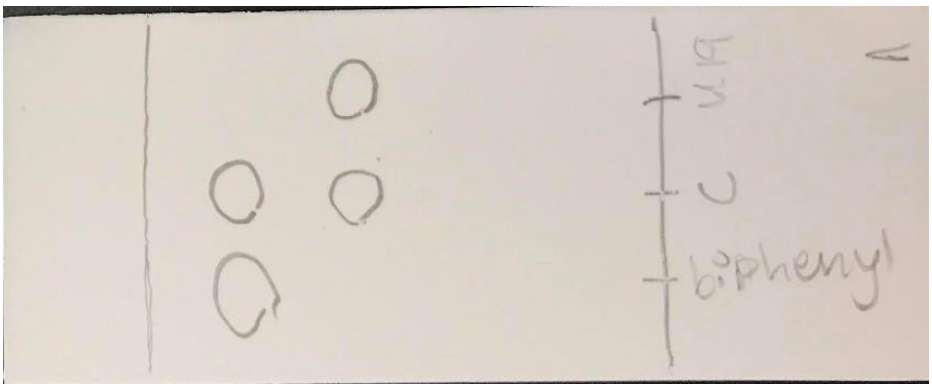
TLC #1



TLC #1
U19 = unknown 19
Benzo = benzophenone
C = co-spot

In solvent of EtOAc : hexane
2 : 8
Rf of Unknown 19= 0.6481
Rf of benzophenone = 0.6574

TLC #2



TLC #2
U19 = unknown 19
C = co-spot

In solvent of EtOAc : hexane
2 : 8
Rf of Unknown 19 = 0.5931
Rf of biphenyl = 0.8029

- Mobile phase smelled of nail polish remover
- The white solid of unknown 19 turned into a colorless liquid in dichloromethane
- Benzophenone and biphenyl are both colorless liquids

Part B : *Effect of Solvent on TLC*

Table of reagents:

Compound	*Molecular Weight (g/mol)*	*Amount*
Ethyl acetate	88.1	10 mL
Hexane	86.0	10 mL
Benzophenone	182.2	2 mL
Biphenyl	154.2	2 mL
Dichloromethane	84.9	2 mL
Unknown 78	unknown	10 mg

Procedure is as described in lab manual (*Experiment 1: Thin Layer Chromatography*. Department of Chemistry. (September 2014). Ottawa: University of Ottawa.)

- Make sure to spot the benzophenone/biphenyl on the left spot before the co- spot (so the capillary tube doesn't get tainted by the unknown on the co-spot position)
- *Unknown 78 was used for this part (Part A showed that unknown 78 is biphenyl)*

TLC #3

TLC #3
U = unknown 78
Biph = biphenyl
C = co-spot

In solvent of EtOAc
Rf of Unknown 78 = 0.8905
Rf of biphenyl = 0.8762

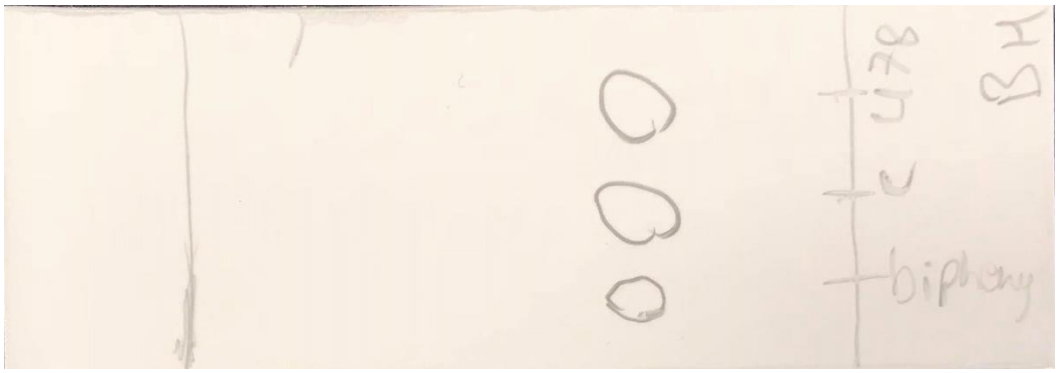
TLC #4

TLC #4
U = unknown 78
Benzo = benzophenone
C = co-spot

In solvent of EtOAc
Rf of Unknown 19 = 0.8942
Rf of benzophenone= 0.8846

- Ethyl acetate (mobile phase) smelled very strongly of nail polish remover, and is a colorless solution
- The white solid of unknown 78 turned into a colorless liquid in dichloromethane

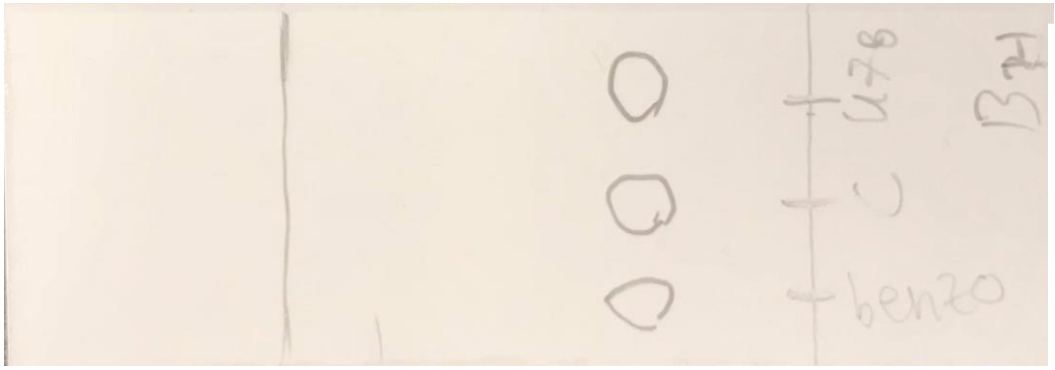
TLC #5



TLC #5
U78 = unknown 78
Bipheny = biphenyl
C = co-spot

In solvent of hexane
Rf of Unknown 78 = 0.3317
Rf of biphenyl = 0.3269

TLC #6



TLC #6
U78 = unknown 78
Benzo = benzophenone
C = co-spot

In solvent of hexane
Rf of Unknown 78 = 0.3333
Rf of benzophenone = 0.3167

- Hexane (mobile phase) smelled slightly of gasoline

Part C : *Ratio of Compounds*

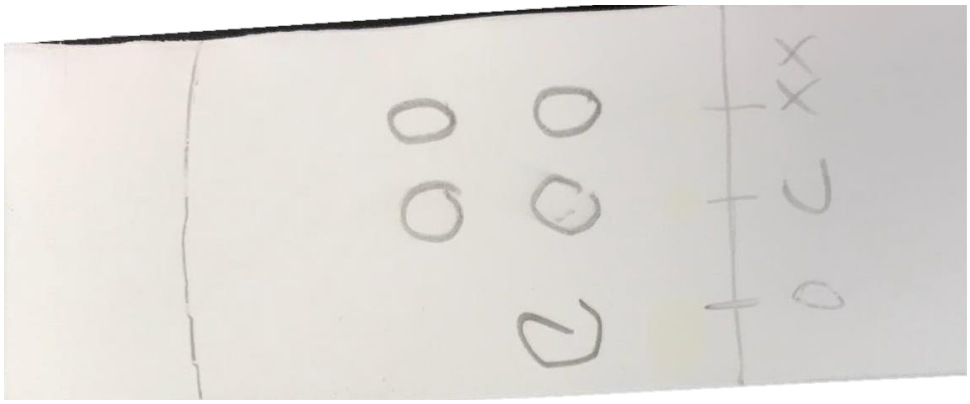
Table of reagents:

Compound	*Molecular Weight (g/mol)*	*Amount*
XX	unknown	2 mL
o-bromonitrobenzene	202.0	1 mL
m- bromonitrobenzene	202.0	1 mL
p- bromonitrobenzene	202.0	1 mL

Procedure is as described in lab manual (*Experiment 1: Thin Layer Chromatography*. Department of Chemistry. (September 2014). Ottawa: University of Ottawa.)

- Make the 10 mL of 9:1 mixture of hexane : ethyl acetate by adding 9 mL of hexane and 1 mL of ethyl acetate in developing jar
- Make sure to spot the benzophenone/biphenyl on the left spot before the co- spot (so the capillary tube doesn't get tainted by the unknown on the co-spot position)

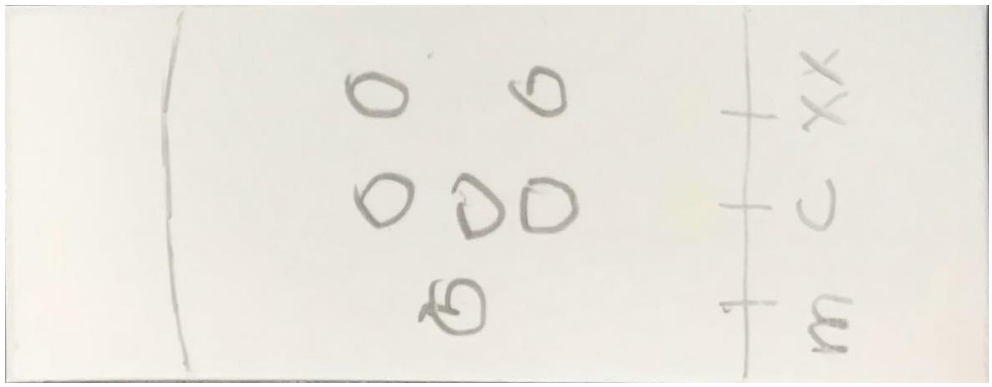
TLC #7



TLC #7
XX = unknown XX
O = ortho-bromonitrobenzene
C = co-spot

In solvent of hexane : EtOAc
9 : 1
Rf 1 of XX = 0.5631
Rf 2 of XX = 0.3050
Rf of O = 0.3191

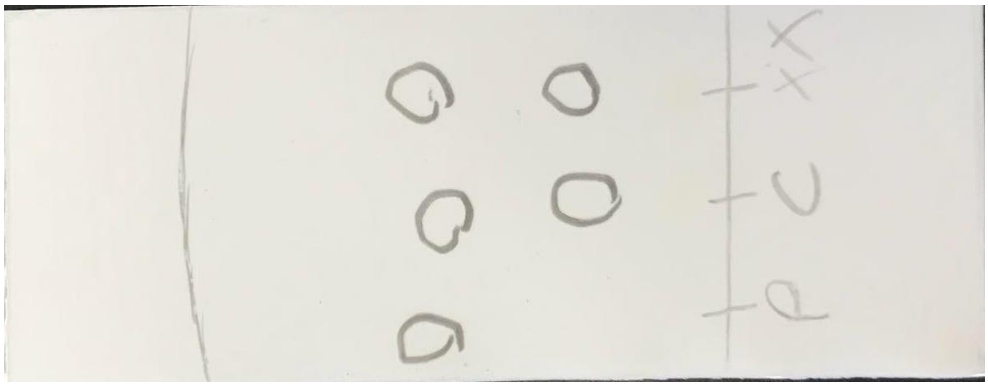
TLC #8



TLC #8
XX = unknown XX
m = meta-bromonitrobenzene
C = co-spot

In solvent of hexane : EtOAc
9 : 1
Rf 1 of XX = 0.6325
Rf 2 of XX = 0.3432
Rf of m = 0.4913

TLC #9



TLC #9
XX = unknown XX
p = para-bromonitrobenzene
C = co-spot

In solvent of hexane : EtOAc
9 : 1
Rf 1 of XX = 0.5744
Rf 2 of XX = 0.2908
Rf of p = 0.5603

- Mobile phase smelled slightly of nail polish remover

- Solution of unknown XX was light yellow in colour
- Solution of ortho-bromonitrobenzene was yellow in colour

CALCULATION:

Calculating % mole of ortho and para isomers in unknown XX

Using TLC #7, it was found that

- Absorbance of ortho-bromonitrobenzene = 19590.158
- Absorbance of para-bromonitrobenzene = 42086.342

- % absorbance of ortho-bromonitrobenzene = 31.8%
- % absorbance of para-bromonitrobenzene = 68.2%

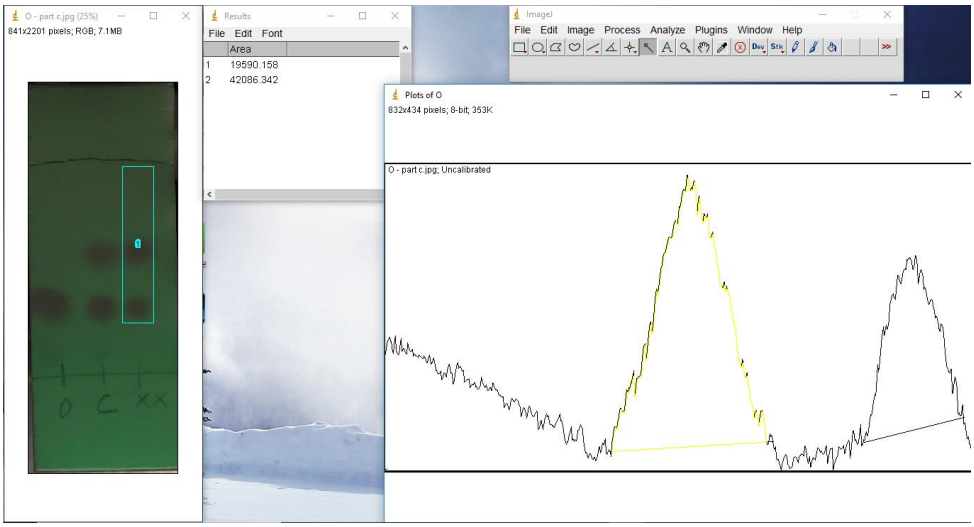
Using calibration curve of "Amount of ortho isomer in an *ortho-para* mixture of bromonitrobenzene"

y = 1.0114x-2.0208 = 31.8%

x = 33.4%

% mole of ortho = 33.4%

% mole of para = 66.6%



DISCUSSION :

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

- In part A of the experiment using the solvent system of ethyl acetate : hexane with a ratio of 2 : 8, it was found out that unknown 19 is benzophenone
- This is because the Rf value of unknown 19 and the Rf value benzophenone are 0.648 and 0.657 respectively (very close in range) and they looked to be on the same row on the silica chromatogram and the co-spot showed only 1 point
- The Rf value of biphenyl was found to be 0.803 which is a lot greater than 0.593 (the Rf value of unknown 19 on the 2nd TLC) and the co-spot showed 2 distinct points, this shows that the unknown is not biphenyl
- The Rf values show that benzophenone is more polar than biphenyl
- The moderate rate of migration of the 3 compounds is due to hydrogen bonding forming between the compound with both the silica and the solvent (although more hydrogen bonds are formed with the silica)

Part B : *Effect of Solvent on TLC*

- For part B, unknown 78 was used (partner's unknown) and from part A it was found that it was biphenyl
- When pure ethyl acetate was used as the solvent, unknown 78 was shown to be both biphenyl and benzophenone, the Rf values of both TLCs were similar (Rf of biphenyl 0.876, Rf of benzophenone 0.885, Rf of unknown 78 0.891 and 0.894) and the co-spot of both TLCs showed to have both 1 point at the top of the TLCs near the solvent front
- The fast rate of migration of biphenyl, benzophenone, and unknown 78 is because the compounds formed hydrogen bonds with the solvent rather than the silica
- When pure hexane was used as the solvent, unknown 78 showed to be both biphenyl and benzophenone similar to using ethyl acetone as the solvent, however the spots were close to the starting line (the Rf of biphenyl 0.327, Rf of benzophenone 0.317, Rf of unknown 78 0.332 and 0.333)
- The slow rate of migration is because the compounds formed hydrogen bonds with the silica only
- Also, because the compounds on a TLC plate in pure hexane/ethyl acetate show little difference in Rf value, this tells us that the difference in polarity between benzophenone and biphenyl is little
- The difference in hydrogen binding is because hexane is much less polar than ethyl acetate

Part C : *Ratio of Compounds*

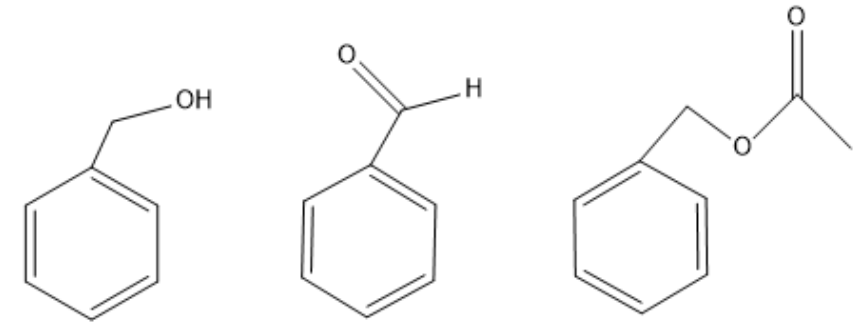
- In this part of the experiment a solvent system of hexane : ethyl acetate with ratio 9 : 1 was used to show the difference in polarity between 3 compounds ortho-bromonitrobenzene, meta-bromonitrobenzene and para-bromonitrobenzene.

- Unknown XX was found to contain ortho-bromonitrobenzene and para-bromonitrobenzene
- This is shown partly in TLC #7 where it is shown that one of the compounds (the bottom, more polar compound) in XX has an Rf value of 0.305 and the Rf value of ortho to be 0.319 with only 2 points on the co-spot (one for the still unknown compound from XX and one from both the 2nd compound in XX and ortho)
- The presence of para-bromonitrobenzene is shown in TLC #9 where the top, less polar compound is shown to have an Rf value of 0.574 while the Rf value of para-bromonitrobenzene is 0.560 and the co-spot also only has 2 points, one contributing to the polar ortho-bromonitrobenzene and the other to the shared (by the unknown XX and the reference para-bromonitrobenzene) para-bromonitrobenzene
- It is shown that there is no meta-bromonitrobenzene because all the Rf values differ greatly and the co-spot shows 3 distinct points, 2 points from the XX and 1 from the meta-bromonitrobenzene
- Using the co-spot values on TLC # 8 and the known values of compounds on XX using TLC #7 and #9, it can be deduced that ortho-bromonitrobenzene is the most polar and para-bromonitrobenzene is the least polar while meta-bromonitrobenzene is in-between

- The % mole of ortho tells us that 33.4% of solution XX is ortho-bromonitrobenzene and 66.6% is para-bromonitrobenzene
- So in any amount of solution XX there will be about 2x more para-bromonitrobenzene than ortho-bromonitrobenzene

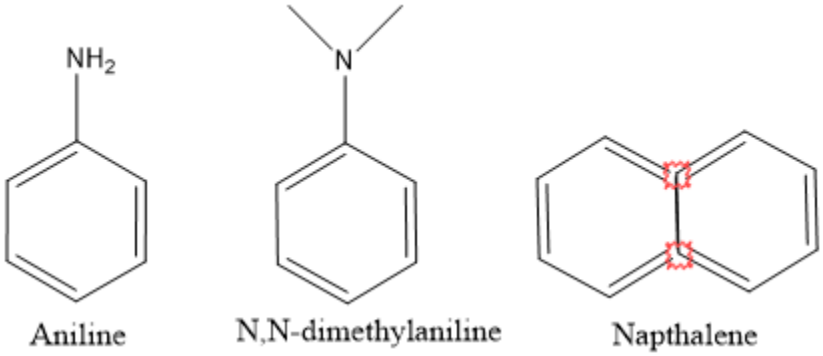
QUESTIONS:

1. Increasing the polarity of the solvent system makes the compounds form more hydrogen bonds with the solvent, causing the compound to migrate across the chromatogram at a faster rate.
2. .
    a. Benyl alcohol is the most polar compound out of these 3, so it will form the most hydrogen bonding with silica, therefore it will be the slowest and be the farthest away from the solvent front, and will have the smallest Rf value.



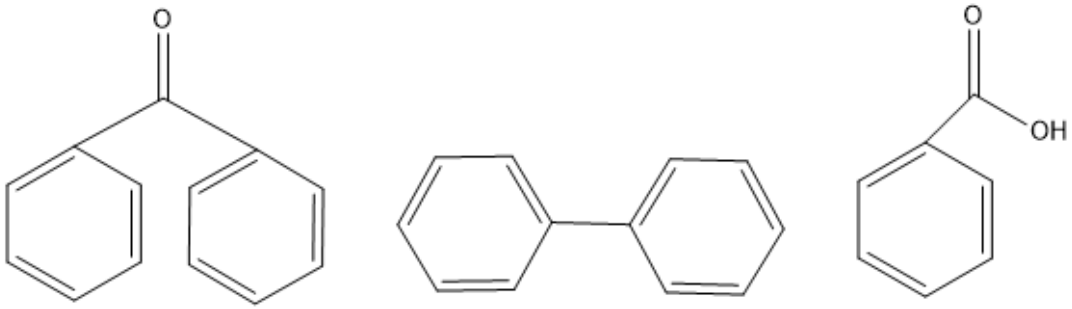
Benzyl Alcohol Benzaldehyde Benzyl Acetate

b. Aniline is the most polar compound out of these 3, it ill form the most H-bonding with silica, it will be the slowest and farthest from the solvent front, and therefore will have the smallest Rf value.



Aniline N,N-dimethylaniline Napthalene

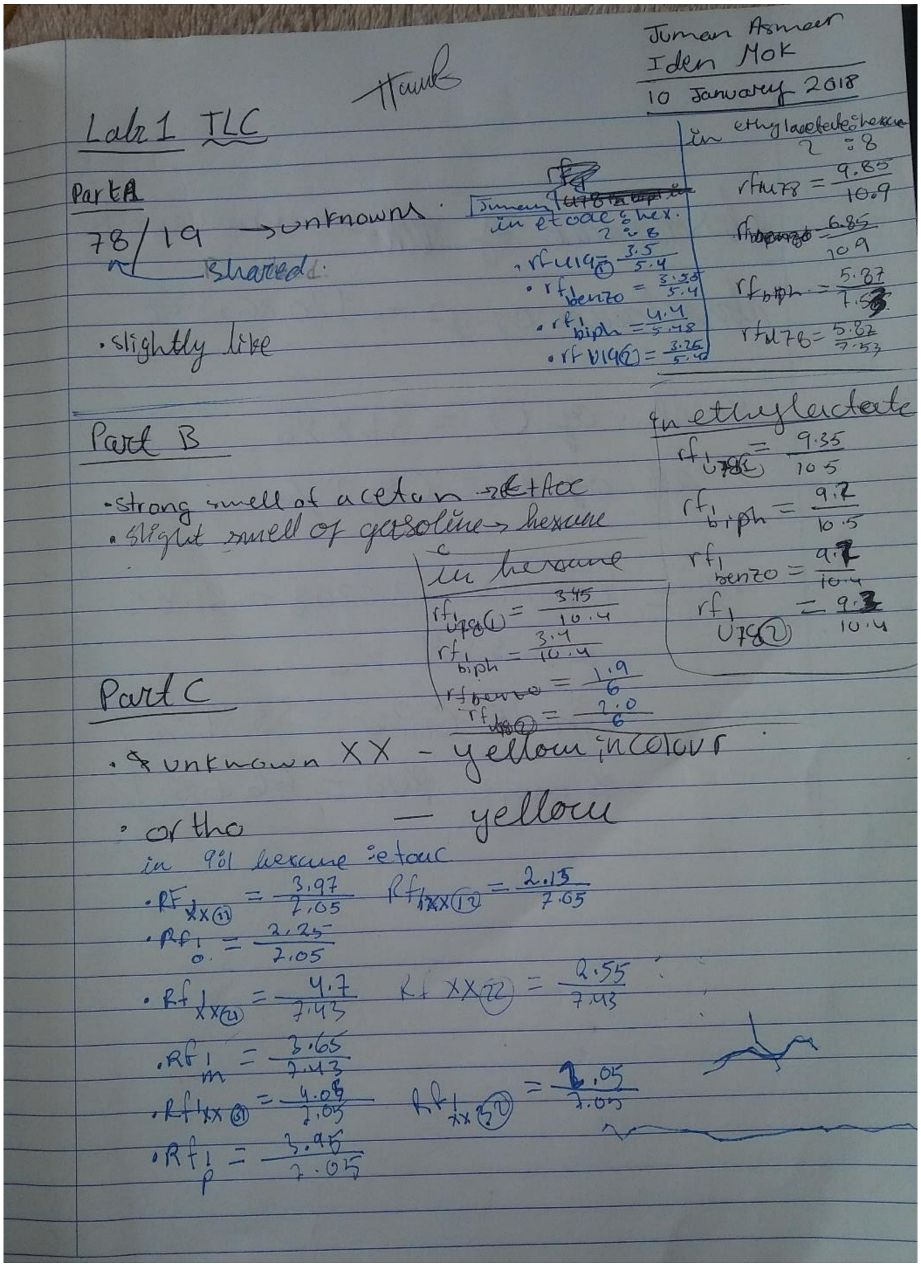
c. Benzoic acid is the most polar compound out of these 3, it will form the most H-bonding with silica, it will be the slowest and farthest from the solvent front, and therefore will have the smallest Rf value.



Benzophenone Biphenyl Benzoic Acid

REFERENCES:

Durst, T., Dr., Scaiano, T., Dr., Ogilvie, W., Dr., & Flynn, A., Dr. (n.d.). *CHM 1321 Organic Chemistry Lab Manual* (pp. 13-19) (B. Van Lierop Dr., A. Bush Mr., & R. Venkateswaran Dr., Eds.



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Iden Mok
10 January 2018

Lab 1 TLC

Part A

78/19 → unknowns
shared

• slightly like

in etoac : hex. 2 : 8
• rf U19(1) = 3.5 / 5.4
• rf benzo = 3.30 / 5.4
• rf biph = 4.4 / 5.48
• rf U19(2) = 3.26 / 5.4

in ethyl acetate : hexane 2 : 8
rf U78 = 9.85 / 10.9
rf = 6.85 / 10.9
rf biph = 5.87 / 7.53
rf U78 = 5.82 / 7.53

Part B

• strong smell of aceton → EtAoc
• slight smell of gasoline → hexane

in hexane
rf U78(1) = 3.45 / 10.4
rf biph = 3.4 / 10.4
rf benzo = 1.9 / 6
rf U19(2) = 2.0 / 6

in ethyl acetate
rf U78 = 9.35 / 10.5
rf biph = 9.2 / 10.5
rf benzo = 9.7 / 10.4
rf U78(2) = 9.3 / 10.4

Part C

• unknown XX – yellow in colour
• ortho – yellow

in 9:1 hexane : etoac
• Rf XX(1) = 3.97 / 7.05 Rf XX(12) = 2.15 / 7.05
• Rf o = 2.25 / 7.05
• Rf XX(21) = 4.7 / 7.43 Rf XX(22) = 2.55 / 7.43
• Rf m = 3.65 / 7.43
• Rf XX(31) = 4.08 / 7.05 Rf XX(32) = 2.05 / 7.05
• Rf p = 3.95 / 7.05