Experiment 1

Experiment Title: Thin Layer Chromatography

Author(s) Name(s): [redacted] 1/

Submitting Author's Partner: [redacted]

TA (Demonstrator)'s Name: [redacted]

Date Experiment Performed: Tuesday, January 14, 2020

Date Experiment Submitted: Tuesday, January 21, 2020

Attach here (if required, indicate the appropriate document(s)):

Medical or other Acceptable Document: _____

Change of Lab Day Form: _____

Change of Lab Section Form: _____

Late Pass: _____

Procedure

1/1

As outlined in the lab manual “Experiment 1: Thin Layer Chromatography”, pages 5-7.

References

Hall, J., Taschuk, M., & Brett, M. (2012). Polarity-adjustable reversed phase ultrathin-layer chromatography. Journal of Chromatography A, 1266, 168–174. https://doi.org/10.1016/j.chroma.2012.10.020

University of Ottawa. “Experiment 1: Thin Layer Chromatography”. Sept 2014.

Observations and Discussion

Part A

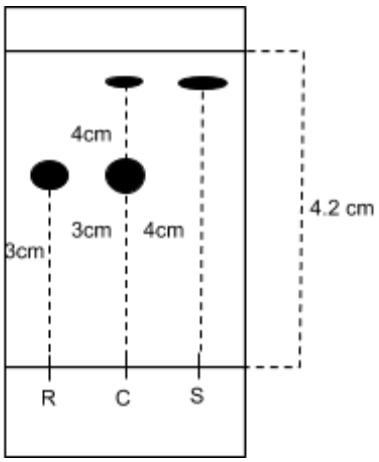
TLC #1

Observations

- Benzophenone - clear, colourless solution
- Biphenyl - clear, colourless solution
- Unknown #47- white powder
- 2:8 EtOAc : Hexanes - clear, colourless solution
- Dichloromethane - clear, colourless solution

12/12 + 4/4

- Developing jar contains 2:8 EtOAc : Hexanes
- R = benzophenone
- C = benzophenone + unknown #47 and dichloromethane
- S = unknown #47 and dichloromethane



By using the calculation found under “Calculations” for the R_f value, the R_f values for TLC #1 are as follows:

Reference:

$R_f = 0.714$

Sample:

$R_f = 0.952$

Co-spot:

$R_f = 0.714$

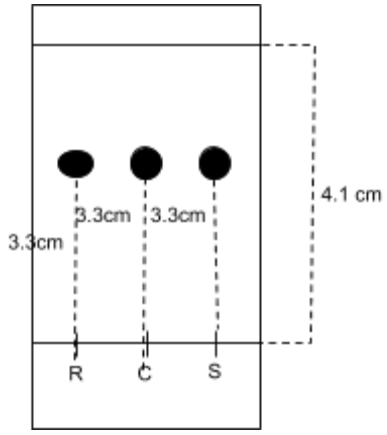
and

$R_f = 0.952$

In TLC plate #1, because the sample spot travelled further up the plate than the reference and had a larger Rf value of 0.952, unknown substance #47 can be concluded to be less polar than benzophenone, which had an Rf value of 0.714. Therefore, unknown substance #47 cannot be identified as benzophenone, nor does it contain benzophenone.

TLC #2

- Developing jar contains 2:8 solution of EtOAc : Hexane
- R = biphenyl
- C = biphenyl + unknown #47 and dichloromethane
- S = unknown #47 and dichloromethane



discussion 4/6

By using the calculation found under calculations for the R_f value, the R_f values for TLC #2 is as follows:

Reference:

$R_f = 0.805$

Sample:

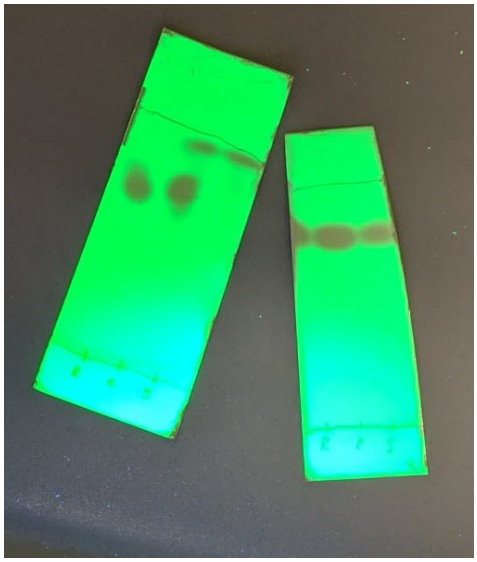
$R_f = 0.805$

Co-spot:

$R_f = 0.805$

xplain why benzophenone is more polar and why biphenyl is less polar

In TLC plate #2, the sample spot has the same Rf value as the reference of 0.805. Therefore, as unknown sample #47 has the same polarity as biphenyl, it can be identified as biphenyl.



TLC plates #1 and #2

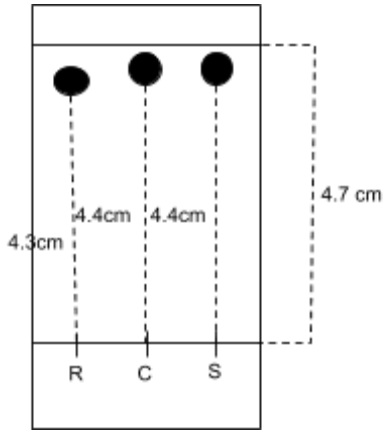
Part B

Observations

- Ethyl acetate - clear, colourless solution
- Hexanes - clear, colourless solution

TLC #1

- Developing jar contains ethyl acetate
- R = benzophenone
- C = benzophenone + unknown #47 and dichloromethane
- S = unknown #47 and dichloromethane



By using the calculation found under calculations for the R_f value, the R_f values for TLC #1 is as follows:

Reference:

$R_f = 0.915$

Sample:

$R_f = 0.936$

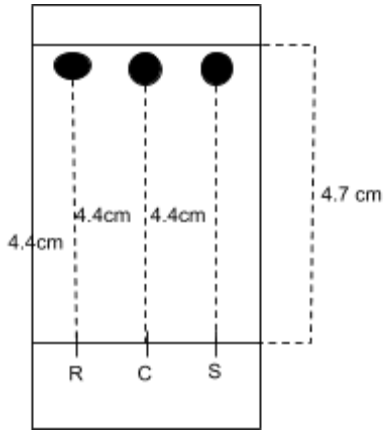
Co-spot:

$R_f = 0.936$

discussion

TLC #2

- Developing jar contains ethyl acetate
- R = Biphenyl
- C = Biphenyl + unknown #47 and dichloromethane
- S = unknown #47 and dichloromethane



By using the calculation found under calculations for the R_f value, the R_f values for TLC #2 is as follows:

Reference:

$R_f = 0.936$

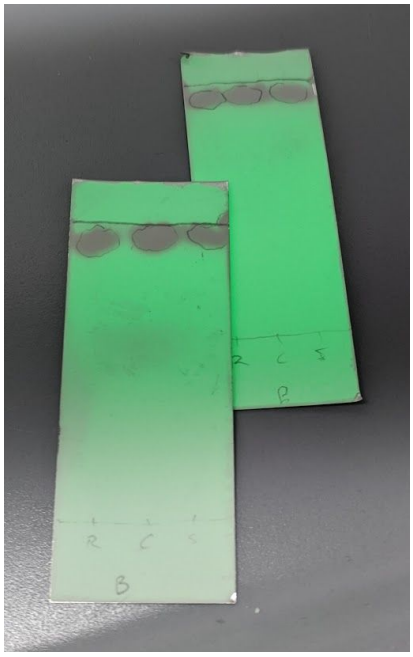
Sample:

$R_f = 0.936$

Co-spot:

$R_f = 0.936$

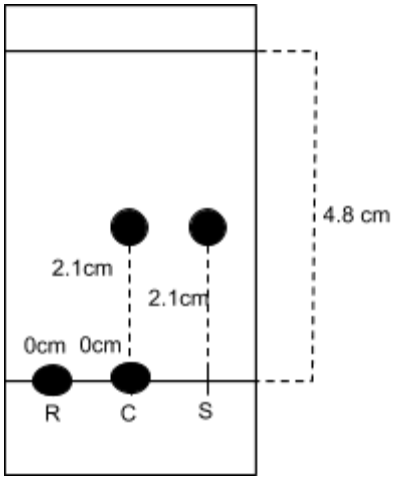
The mobile phase containing only EtOAc is much more polar than that used in Part A, 2:8 EtOAc:Hexanes. A more polar solvent causes the substances to travel further up the plate, resulting in higher Rf values, as it disrupts the forces between these compounds and the silica gel plate. This is evident as the Rf values of the same substances in EtOAc are greater than those in Part A, in a less polar solvent. This mobile phase is not an effective solvent to use because there is not a good separation between the substances.



TLC plates #1 and #2

TLC #3

- Developing jar contains hexanes
- R = Benzophenone
- C = Benzophenone + unknown #47 and dichloromethane
- S = unknown #47 and dichloromethane



By using the calculation found under calculations for the R_f value, the R_f values for TLC #3 is as follows:

Reference:

$R_f = 0$

Sample:

$R_f = 0.438$

Co-spot:

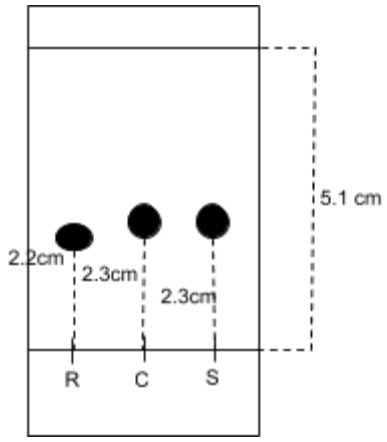
$R_f = 0$

and

$R_f = 0.438$

TLC #4

- Developing jar contains hexanes
- R = Biphenyl
- C = Biphenyl + unknown #47 and dichloromethane
- S = unknown #47 and dichloromethane



By using the calculation found under calculations for the R_f value, the R_f values for TLC #4 is as follows:

Reference:

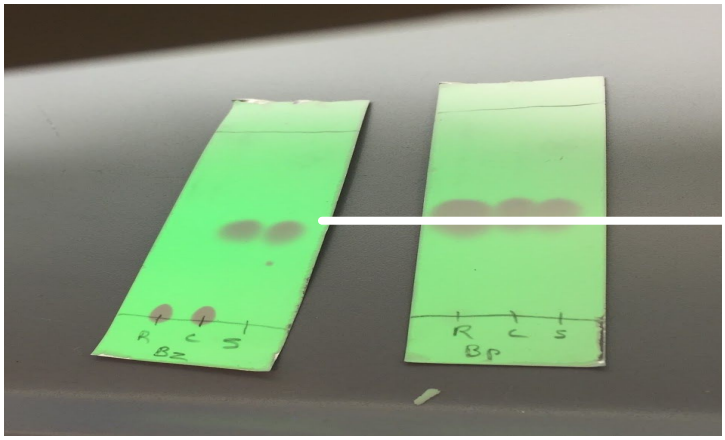
$R_f = 0.431$

Sample:

$R_f = 0.451$

Co-spot:

$R_f = 0.451$



TLC plates #3 and #4

A mobile phase containing only hexanes is low in polarity. It is not very effective in eluting the substances from the silica gel, so the substances do not travel as far up the plate. Because benzophenone is quite polar, it did not travel up the plate at all and has an Rf value of 0. The Rf values of the same substances with hexanes as the mobile phase are less than those in Part A, with a more polar mobile phase.

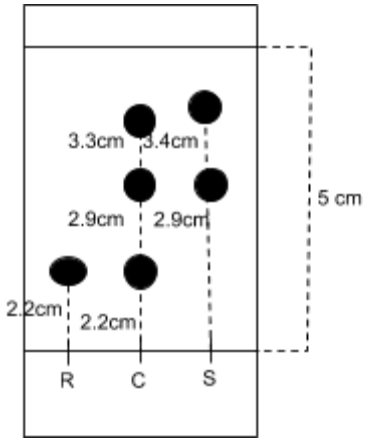
Part C

Observations

- 9:1 Hexanes : EtOAc - clear, colourless solution
- *o*-bromonitrobenzene - clear, colourless solution
- *m*-bromonitrobenzene - clear, colourless solution
- *p*-bromonitrobenzene - clear, colourless solution
- Unknown “k” - clear, colourless solution

TLC #1

- Developing jar contains 9:1 solution of hexane : EtOAc
- R = *o*-bromonitrobenzene
- C = *o*-bromonitrobenzene + unknown “k”
- S = unknown “k”



By using the calculation found under calculations for the R_f value, the R_f values for TLC #1 is as follows:

Reference:

$R_f = 0.44$

Sample:

$R_f = 0.58$

and

$R_f = 0.68$

Co-spot:

$R_f = 0.44$

and

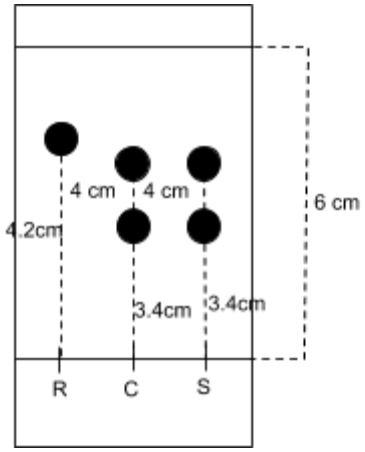
$R_f = 0.58$

and

$R_f = 0.66$ 3/3

TLC #2

- Developing jar contains 9:1 solution of hexane : EtOAc
- R = *m*-bromonitrobenzene
- C = *m*-bromonitrobenzene + unknown “k”
- S = unknown “k”



By using the calculation found under calculations for the R_f value, the R_f values for TLC #2 is as follows:

Reference:

$R_f = 0.70$

Sample:

$R_f = 0.57$

and

$R_f = 0.667$

Co-spot:

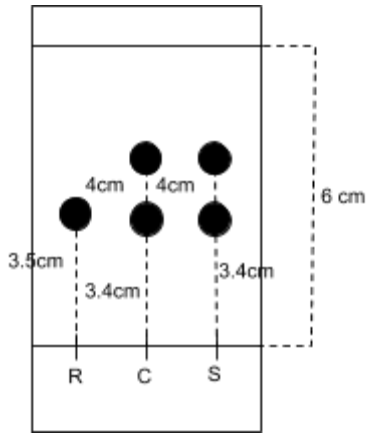
$R_f = 0.57$

and

$R_f = 0.667$

TLC #3

- Developing jar contains 9:1 solution of hexane : EtOAc
- R = *p*-bromonitrobenzene
- C = *p*-bromonitrobenzene + unknown “k”
- S = unknown “k”



By using the calculation found under calculations for the R_f value, the R_f values for TLC #3 is as follows:

Reference:

$R_f = 0.583$

Sample:

$R_f = 0.567$

and

$R_f = 0.667$

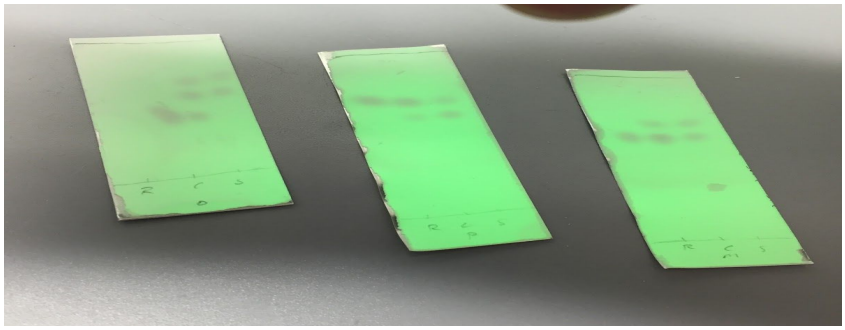
Co-spot:

$R_f = 0.567$

and

$R_f = 0.667$

TLC plates #1,2, and 3



Far right TLC plate had a scratch in it (the lowest mark in the s column)

Calculations

$R_f = \frac{d_1}{d_s}$

1/1 Samples:

Part A, TLC #1 reference:

$R_f = \frac{3\ cm}{4.2\ cm}$

$= 0.714$

Part A, TLC #1 sample:

$R_f = \frac{4\ cm}{4.2\ cm}$

$= 0.952$

Part A, TLC #1 co-spot:

$R_f = \frac{3\ cm}{4.2\ cm}$

$= 0.714$

and

$R_f = \frac{4\ cm}{4.2\ cm}$

$= 0.952$

Unknown “k” contains two of the three compounds given (*m*-bromonitrobenzene, *p*-bromonitrobenzene, *o*-bromonitrobenzene). As supported by the TLC plates, the unknown sample “k” gives two distinct spots. As seen in TLC plate #3, the R_f value of *p*-bromonitrobenzene of 0.70 is quite close to that of one of the two spots of the unknown sample that travelled further up the plate, 0.667. Therefore, this spot (which is less polar than the other spot) is identified to be *p*-bromonitrobenzene.

As seen in TLC plate #2, the Rf value of *m*-bromonitrobenzene of 0.583 is quite close to that of the other spot of the unknown sample, 0.567. Therefore, this spot (which is more polar than the other spot) is identified to be *m*-bromonitrobenzene.

In TLC plate #1, *o*-bromonitrobenzene resulted in an Rf value of 0.44. As this is not similar to the Rf values given by either spot of the unknown sample, it is concluded that the unknown sample does not contain *o*-bromonitrobenzene.

Conclusion

The unknown substance #47 for part A is assumed to be biphenyl due to polarities being the same (as shown in the TLC plates). As shown by the results in part B, a more polar mobile phase causes the substances to travel further up the plate. Conversely, a less polar mobile phase causes the substances to not travel as far up the plate. The unknown substance “k” for part C is assumed contain *m*-bromonitrobenzene and *p*-bromonitrobenzene as concluded by the Rf values.

Questions

1.

- Important to make co-spots last to prevent the contamination of materials
- If the co-spots were made first and the same capillary tube was used again, spots made afterwards would be contaminated as the capillary tube came into contact with another substance
- Making co-spots last ensures that the spots on their respective lanes are as uncontaminated as possible to maximize the accuracy of results

1/1

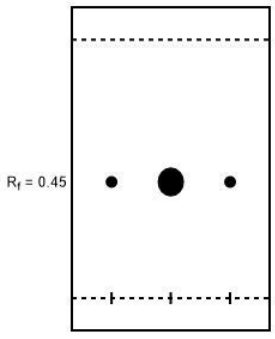
2.

- Mobile phase and stationary phase compete to attract compound
- Polar compounds more strongly attracted to solvent system of increased polarity, moving materials further up plate
- More polar solvents (mobile phase) can better elute materials from plate (stationary phase)
- R_f values for all materials increases
- Less polar materials travel further than more polar materials

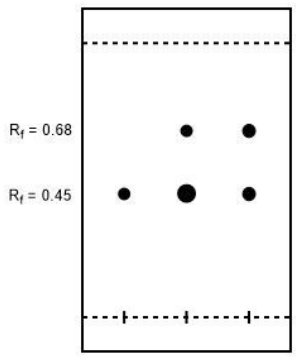
1/2

3.

a.



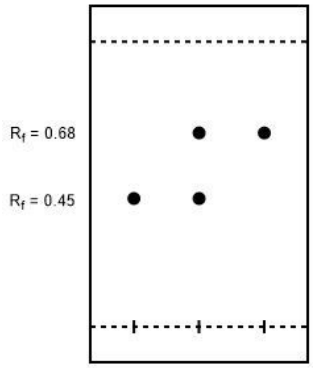
b.



6/

question

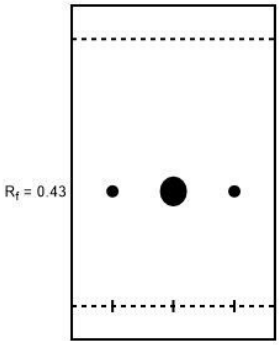
c.



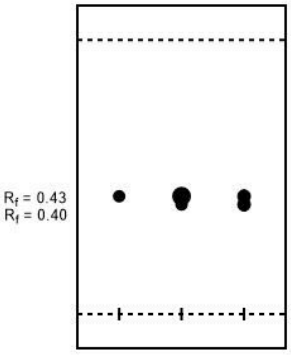
4.

a.

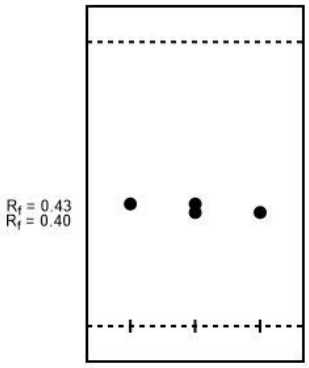
6/6



b.



c.

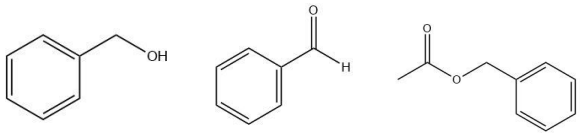


5.

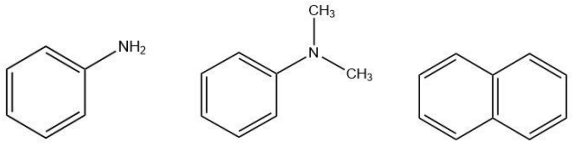
a.

i. Benzyl alcohol, benzaldehyde, benzyl acetate

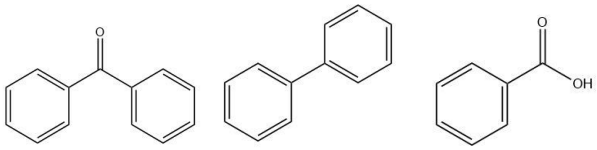
7/



ii. Aniline, *N,N*-dimethylaniline, naphthalene



iii. Benzophenone, biphenyl, benzoic acid



b.

aldehyd este

i. Benzyl acetate, ~~benzaldehyde~~, benzyl alcohol

ester has two

ii. Naphthalene, *N,N*-dimethylaniline, aniline

iii. Biphenyl, benzophenone, benzoic acid

c.

i. The net dipole-dipole force for benzyl acetate is lower than the rest of the molecules, this results in it being the least polar. The net dipole-dipole force for benzaldehyde adds up to a higher number, causing it to be more polar than benzyl acetate however less polar than benzyl alcohol due to benzyl alcohol's OH^- group. The OH^- group on benzyl acetate, makes the molecule more polar than the other two due to the ability to hydrogen bond. The benzyl alcohol also has a smaller R_f value than both of the other molecules.

ii. Naphthalene is a symmetrical molecule, this yields for the dipole-dipole forces to be canceled out by one another. This causes a neutral net dipole force and a nonpolar molecule. N,N-dimethylaniline has a net dipole force lower than that of aniline. Aniline also has the ability to hydrogen bond which also result in aniline molecule being more polar.

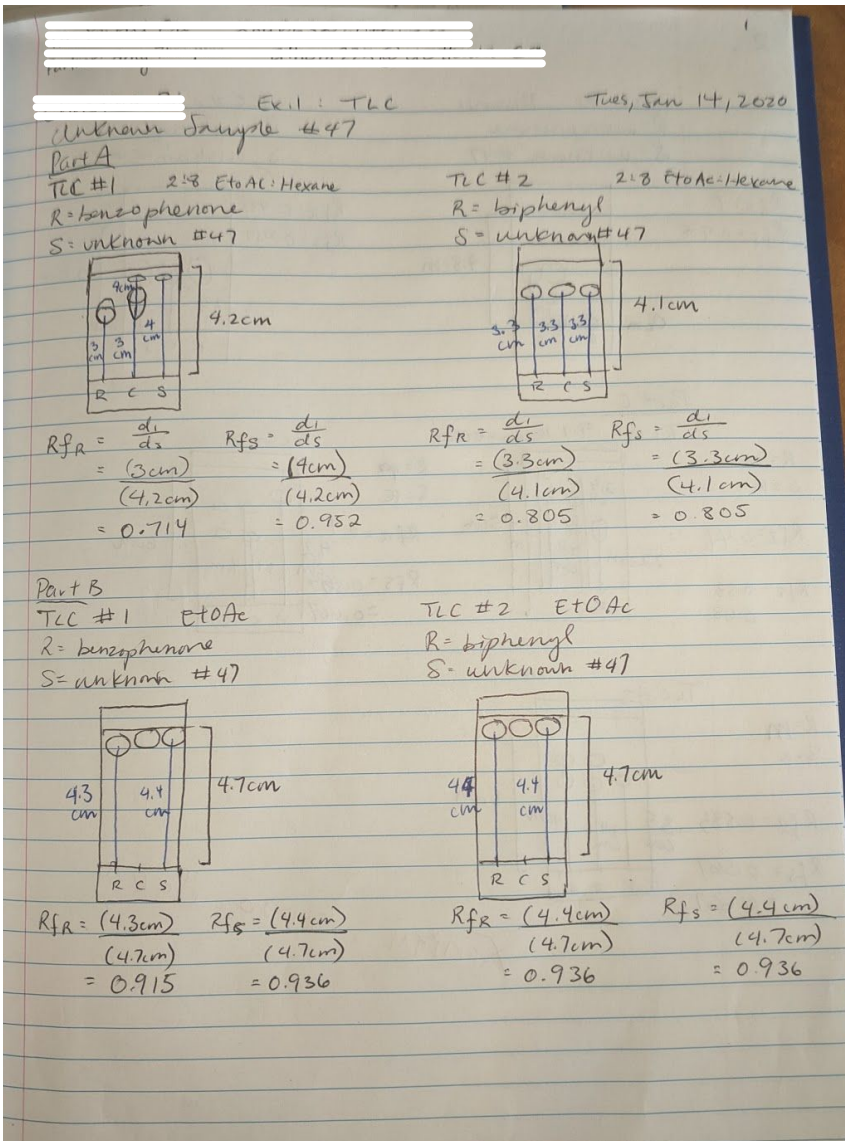
iii. Biphenyl is a symmetrical molecule which results in the dipole-dipole forces to be canceled out by each other and the molecule to be nonpolar. Benzophenone results in a lower dipole-dipole force than benzoic acid. Benzoic acid has the smallest R_f value and also contains an OH^- group

which is capable of hydrogen bonding. This yields for the benzoic acid being the most polar molecule out of the three.

Sources of Error

- Through physical contact while handling the silica gel plates and use of capillary tube, the disruption of the silica coating could affect the movement of substances on plate
- Spotting using capillary tubes results in inconsistent spot sizes, which could affect results
- Hand-drawn solvent front, base lines, and compound lanes are not exactly straight, thus possibly affecting accuracy of measurements used for R_f value calculations
- Highly volatile solvent evaporating before solvent front is drawn could affect measurements used for R_f value calculations

Raw Data



[redacted]

Ex. 1 : TLC — Tues, Jan 14, 2020

Unknown Sample #47

Part A

TLC #1 — 2:8 EtOAc : Hexane

R = benzophenone

S = unknown #47

$Rf_R = \frac{d_1}{d_2} = \frac{(3cm)}{(4.2cm)} = 0.714$

$Rf_S = \frac{d_1}{d_2} = \frac{(4cm)}{(4.2cm)} = 0.952$

TLC #2 — 2:8 EtOAc : Hexane

R = biphenyl

S = unknown #47

$Rf_R = \frac{d_1}{d_2} = \frac{(3.3cm)}{(4.1cm)} = 0.805$

$Rf_S = \frac{d_1}{d_2} = \frac{(3.3cm)}{(4.1cm)} = 0.805$

Part B

TLC #1 — EtOAc

R = benzophenone

S = unknown #47

$Rf_R = \frac{(4.3cm)}{(4.7cm)} = 0.915$

$Rf_S = \frac{(4.4cm)}{(4.7cm)} = 0.936$

TLC #2 — EtOAc

R = biphenyl

S = unknown #47

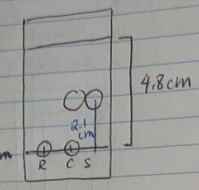
$Rf_R = \frac{(4.4cm)}{(4.7cm)} = 0.936$

$Rf_S = \frac{(4.4cm)}{(4.7cm)} = 0.936$

2

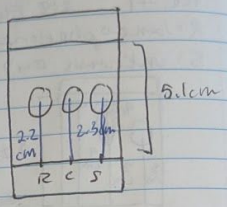
TLC #1 Hexanes
R = benzophenone
S = unknown #47

$R_f R = 0$
$R_f S = 0.438$



TLC #2 Hexanes
R = biphenyl
S = unknown #47

$R_f R = 0.431$
$R_f S = 0.451$



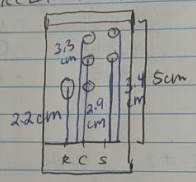
Part C

TLC #1 9:1 Hexane-EtOAc

R = 0
S = IC

$R_f R = 0.44$

$R_f S = 0.58$
0.68

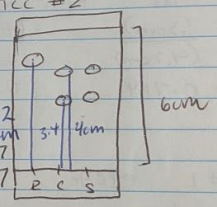


TLC #2

R = ~~pp~~
S = IC

$R_f R = 0.7$

$R_f S = 0.567$
$= 0.667$

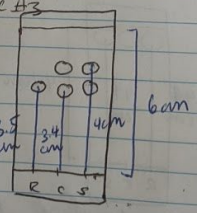


TLC #3

R = ~~pp~~
S = IC

$R_f R = 0.583$

$R_f S = 0.567$
$= 0.667$



2020 Jan 14

①

[redacted]

January 14, 2020

TA: [redacted]

unknown compound # 47

Experiment 1

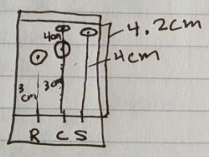
Part A

TLC #1 2:8 EtOAc: Hexane

R = benzophenone

C = co-spot

S = unknown #47 and dichloromethane



$R_{fR} = \frac{d_1}{d_3} = \frac{3cm}{4.2cm} = 0.714$

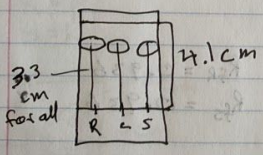
$R_{fS} = \frac{d_1}{d_3} = \frac{4cm}{4.2cm} = 0.952$

TLC #2 2:8 EtOAc: Hexane

R = Biphenyl

C = CO-spot

S = unknown #47 and dichloromethane



$R_{fR} = \frac{d_1}{d_5} = \frac{3.3cm}{4.1cm} = 0.805$

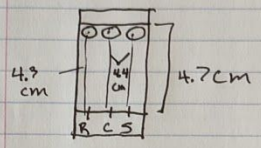
$R_{fS} = \frac{d_1}{d_5} = \frac{3.3cm}{4.1cm} = 0.805$

(2)

Part B

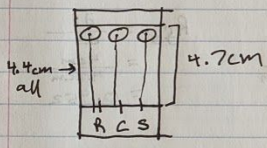
Jan 14

TLC #1 ethyl acetate
R = Benzophenone
S = #47 unknown

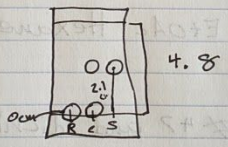


$R_{fR} = 0.914$ $R_{fR} = 0.936$

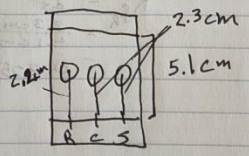
TLC #2 ethyl acetate
R = Biphenyl
S = #47 unknown



TLC #3 Hexanes
R = Benzophenone
S = #47 unknown



TLC #4 Hexanes
R = Biphenyl
S = #47 unknown



$R_{fR} = 0.936$
$R_{fS} = 0.936$

(3)

Jan 14

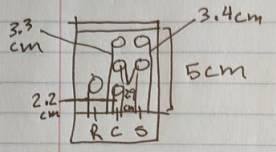
Part C

↳unknown = K

TLC #1 9:1 Hexane : EtOAc

R = O

S = K



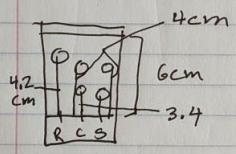
R_{fR} = 0.44

R_{fS} = 0.58 and 0.68

TLC #2 9:1 H : E

R = m

S = K



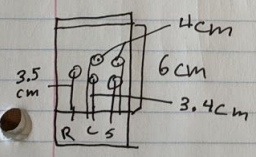
R_{fR} = 0.7

R_{fS} = 0.567 and 0.667

TLC #3 9:1 H : E

R = P

S = K



R_{fR} = 0.583

R_{fS} = 0.567 and 0.667

2020 Jan 14