CHM1321 LAB 1: Thin Layer Chromatography

CHM1321 D

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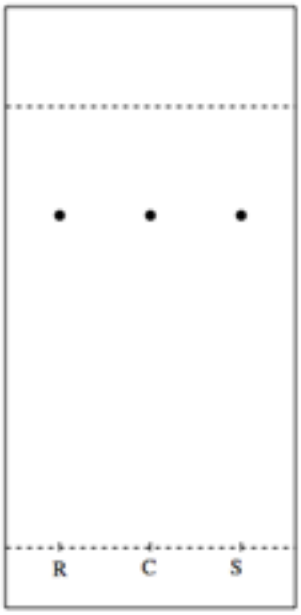
Observations and Results

PART A

Our unknown compound was number 57. The compound was white crystals that resembled course salt and it had a rubber odour. Our sample was compound #57 that completely dissolved when mixed with dichloromethane (clear liquid). The Ethyl Acetate : Hexanes 2:8 developer (solvent) was a clear liquid that had an odour similar to that of nail polish remover. The benzophenone (reference #1) was a clear liquid. Biphenyl (reference #2) was a clear liquid that had an odour resembling that of outdoor house paint.

Once the TLC plates were removed from the developer and dried and analyzed under the UV light, the TLC plate #1 with the benzophenone as the reference had three dots all in a straight line on the plate (same Rf). TLC plate #2 with the biphenyl reference had two dots that were high (one from the reference lane and one from the co-spot lane) and two dots just below those ones (one dot from the co-spot lane and the second from the sample lane).

Part A TLC #1



Developer:
The Ethyl Acetate : Hexanes 2:8

Rf: 0.73

Sample:
Compound #57

Reference:
benzophenone

Part A TLC #2

Developer:
The Ethyl Acetate : Hexanes 2:8

Rf: 0.69 and 0.91

Sample:
Compound #57

Reference:
biphenyl

R: Reference
C: Co-spot
S: Sample

PART B

Our developer/solvent #1 was Ethyl Acetate which is a clear liquid and smells like nail polish. Developer/eluant #2 is Hexane which is a clear liquid and smells like wood polish. The references were again benzophenone and biphenyl and our sample was the same sample as in Part A.

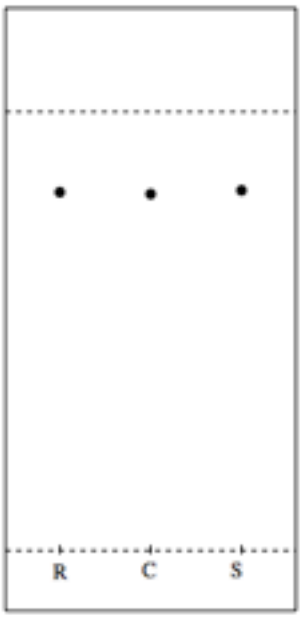
Results in the Ethyl Acetate as the developer:

TLC plate #1 with benzophenone as the reference had the same result as in Part A. The three dots from each lane where in a horizontal line on the same level on the plate. TLC plate #2 with the biphenyl as the reference had all three dots from each lane in a straight horizontal line in the same level. The three dots were high up the plate right below the solvent line. Both plates had the exact same results.

Results in the Hexane as the developer:

TLC plate #1 with the benzophenone as the reference had three dots on the same horizontal line, yet they were located just above the line distinguishing the lanes, hence located near bottom of plate. TLC plate #2 with the biphenyl reference had two dots located near the bottom of the plate (one from the reference lane one from the co-spot lane) and two dots midway up the plate (one from the co-spot lane and on dot from the sample lane).

Part B TLC #1 EA



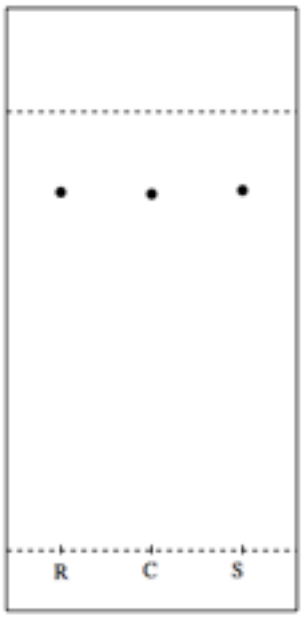
Developer:
Ethyl Acetate

Rf: 0.90

Sample:
Compound #57

Reference:
Benzophenone

Part B TLC #2 EA



Developer:
Hexanes

Rf: 0.93

Sample:
Compound #57

Reference:
Biphenyl

Part B TLC #1 HEX

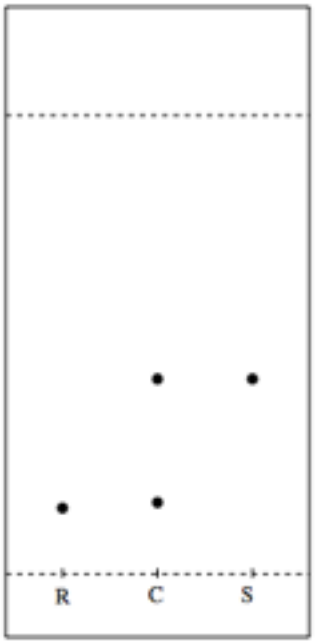
Developer:
Hexanes

Rf: 0.2

Sample:
Compound #57

Reference:
Benzophenone

Part B TLC #2 HEX



Developer:
Hexanes

Rf: 0.2 and 0.43

Sample:
Compound #57

Reference:
Biphenyl

PART C

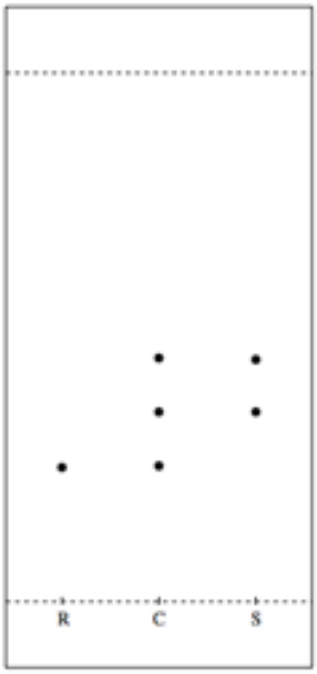
Our unknown compound was YY which was a clear liquid that smelled like window cleaner. Our references were: O-bromonitrobenzene, that was a transparent liquid with a yellow hue. M-bromonitrobenzene, and P-bromonitrobenzene, that were clear transparent liquids. Our developer was 9:1 Hexanes: Ethyl Acetate which was a clear liquid.

The TLC Plate O which had O-bromonitrobenzene as our reference had two dots on the same level nearest to the bottom of the plate (one from the reference lane and one dot from the co-spot lane). There is a row of dots above the previous bottom-level and in the middle of the plate (one dot from the co-spot and one from the sample lane). There is a row of dots above the previous mid-level (one from the co-spot lane and one dot from the sample lane).

The TLC plate M which had M-bromonitrobenzene as the reference had a row of three dots in a line high up the plate (one from the reference lane, one dot from the co-spot lane and one dot from the sample lane). There was a row of two dots above the previous level of dots (one from the co-spot lane and one dot from the sample lane).

The TLC plate P which had P-bromonitrobenzene as the reference had a row of two dots in a line mid-way up the plate (one dot from the co-spot lane and one dot from the sample lane). There was a row of three dots above the previous mid-level row of dots (one dot each from the reference, co-spot and sample lanes)

Part C TLC “O”



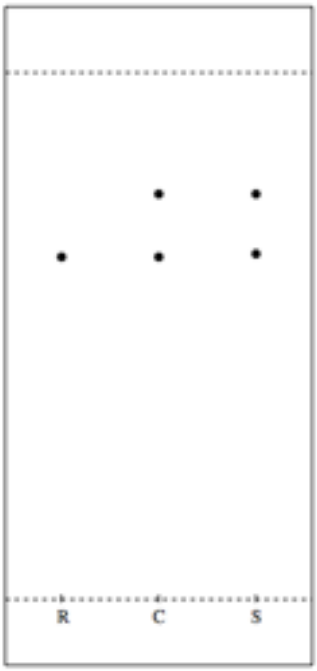
Developer:
9:1 Hexanes: Ethyl Acetate

Rf: 0.59 and 0.46 and 0.33

Sample:
Compound YY

Reference:
O-bromonitrobenzene

Part C TLC “M”



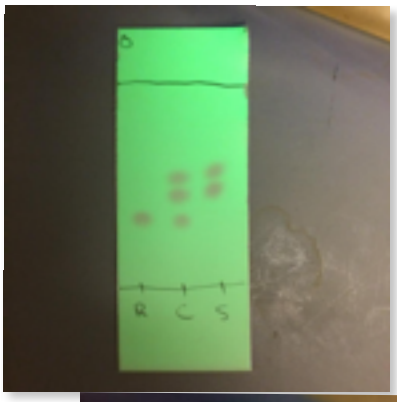
Developer:
9:1 Hexanes: Ethyl Acetate

Rf: 0.56 and 0.48

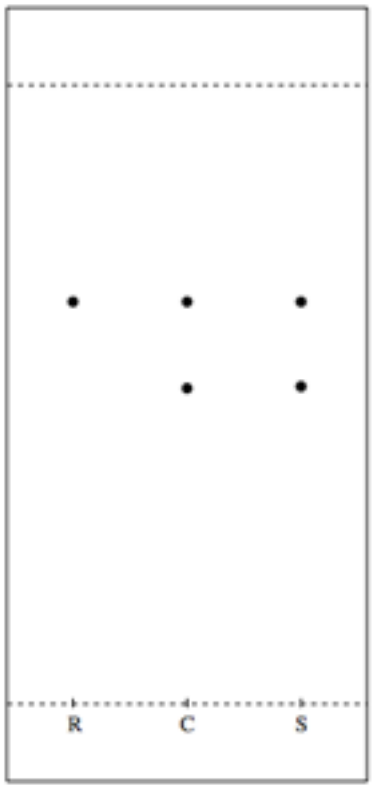
Sample:
Compound YY

Reference:
M-bromonitrobenzene

Part C TLC “O”



Part C TLC “P”



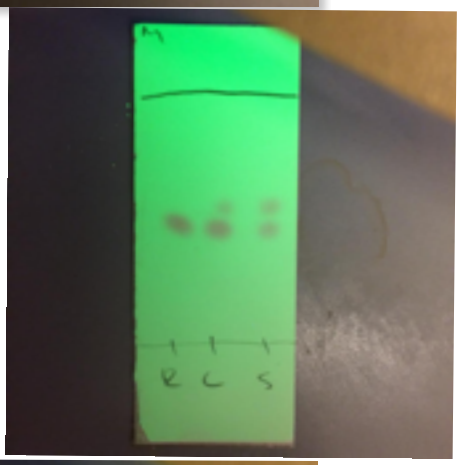
Developer:
9:1 Hexanes: Ethyl Acetate

Rf: 0.52 and 0.38

Sample:
Compound YY

Reference:
P-bromonitrobenzene

Part C TLC “M”



Part C TLC “P”

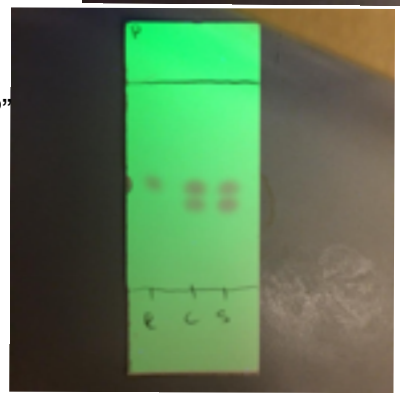


Table 1: Data from PART A **(Rf values are bolded)**

TLC plates	Reference	Distance Travelled by Solvent (cm)	Sample (cm) from start line	Co-spot from start line (cm)	Reference from start line (cm)
1	Benzophenone	4.9	3.6 **(0.73)**	3.6 and 3.6 **(0.73)**	3.6 **(0.73)**
2	Biphenyl	4.5	3.1 **(0.69)**	3.1 **(0.69)** and 4.1 **(0.91)**	4.1 **(0.91)**

Table 2: Data from PART B **(Rf values Bolded)**

TLC plates	Developer/ solvent	Reference	Distance Travelled by Solvent (cm)	Sample (cm) from start line	Co-spot from start line (cm)	Reference from start line (cm)
1 EA	Ethyl Acetate	Benzophenone	4.2	3.8 **(0.90)**	3.8 **(0.90)** and 3.8 **(0.90)**	3.8 **(0.90)**
2 EA	Ethyl Acetate	Biphenyl	4.3	4 **(0.93)**	4.0 **(0.93)** and 4.0 **(0.93)**	4 **(0.93)**
3 HEX	Hexanes	Benzophenone	4.4	0.1 **(0.02)**	0.1 **(0.02)**	0.1 **(0.02)**
4 HEX	Hexanes	Biphenyl	4.4	1.9 **(0.43)**	1.9 **(0.43)** and 0.1 **(0.02)**	0.1 **(0.02)**

Table 3: Data from PART C **(Rf values Bolded)**

TLC plates	Reference	Distance Travelled by Solvent (cm)	Sample (cm) from start line	Co-spot from start line (cm)	Reference from start line (cm)
"O"	O-bromonitrobenzene	3.9	2.3 **(0.59)** and 1.8 **(0.46)**	2.3 **(0.59)** and 1.8 **(0.46)** and 1.3 **(0.33)**	1.3 **(0.33)**
"M"	M-bromonitrobenzene	3.9	2.2 **(0.56)** and 1.9 **(0.48)**	2.2 **(0.56)** and 1.9 **(0.48)**	1.9 **(0.48)**
"P"	P-bromonitrobenzene	3.8	2.0 **(0.52)** and 1.5 **(0.38)**	2.0 **(0.52)** and 1.5 **(0.38)**	1.5 **(0.38)**

Sample Calculations

Part A

TLC plate #1 w/ benzophenone:

Solvent line: 4.9 cm
Sample: 3.6 cm
Co-spot: 3.6 cm
Reference: 3.6 cm

Rf = Distance of sample / distance of solvent line
Rf = 3.6 cm / 4.9 cm
Rf = 0.73

Rf = Distance of co-spot / distance of solvent line
Rf = 3.6 cm / 4.9 cm
Rf = 0.73

Rf = Distance of reference / distance of solvent line
Rf = 3.6 cm / 4.9 cm
Rf = 0.73

TLC plate #2 w/ biphenyl

Solvent line: 4.5 cm
Sample: 3.1 cm
Co-spot: 3.1 cm and 4.1
Reference: 4.1 cm

Rf = Distance of sample / distance of solvent line
Rf = 3.1 cm / 4.5 cm
Rf = 0.69

Rf = Distance of co-spot / distance of solvent line
Rf = 3.1 cm / 4.5 cm
Rf = 0.69

Rf = Distance of co-spot / distance of solvent line
Rf = 4.1 cm / 4.5 cm
Rf = 0.91

Rf = Distance of reference / distance of solvent line
Rf = 4.1cm / 4.5 cm
Rf =0.91

Calculating Percentage Peak Intensity and Ratio

Using the ImageJ

% of peak 1 of lane 3 in M-bromonitrobenzene TLC plate:
100%

$Y=-0.0127x^2+1.9753x+1.1565$
$Y= -0.0127(100)^2+1.9753(100)+1.1565$
Y= -127 + 197.53 + 1.1565
Y= 71.6865 %

Compound YY is composed of 71.7% M-bromonitrobenzene

Mole % of YY compound= 100%-71.7% = 28.3% of P-bromonitrobenzene

Ratio of M-bromonitrobenzene : P-bromonitrobenzene
2.5: 1

Discussion

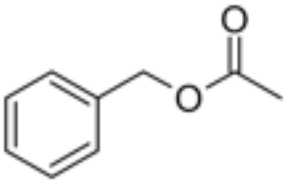
For Part A, because TLC plate #1 had all dots in the same line, this means that our sample must contain benzophenone, because the sample and the reference dots all have the same Rf = 0.73 (which is the measurement of the polarity of the compound).

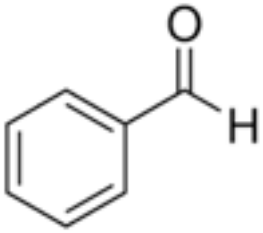
TLC plate #2 showed that the reference and the sample dots had different Rf, thus they have different polarities, thus the sample cannot be composed of biphenyl.
For Part B, Ethyl Acetate might have been too polar for the molecules used in this part because both plates showed the same results. For both plates, all dots showed in a straight line on the same level, right below the solvent line. This means the solvent/ developer (Ethyl Acetate) was much too polar and the compounds where mostly attracted to the solvent rather than the silica gel (absorbent). Thus when the plate was dipped in the solvent is brought the compounds all the way up the plate with it, not letting the compounds stick to the plate. Therefore the plates used in the Ethyl Acetate developer are inaccurate results.
For Part C, we know that YY is composed of two molecules because it produces two dots on the sample lane on the TLC Plates. We know from Part C that the compound YY is made up of M-bromonitrobenzene and P-bromonitrobenzene because at least one of the dots of the YY compound lined up with the dots of these compounds on the TLC plate. Also the reference dots have the same Rf value as the sample dots (0.56 and 0.48 for M and 0.52 and 0.38 for P). None of the YY compound dots lined with the O-bromonitrobenzene reference dot, thus this compound is not found in the YY compound.Also none of the reference dots had matching Rf values with the sample dots, thus justifying that compound YY is not composed of O-bromonitrobenzene. The ratio of the YY compound is 2.5: 1 M-bromonitrobenzene : P-bromonitrobenzene. 71.7 % of the compound was M-bromonitrobenzene while 28.3% of the compound was P-bromonitrobenzene.
Examples of errors conducted in this lab would include adding too little of drops onto the TLC plate, which would cause no dots too appear on the TLC plate causing invalid results. Also exposing the whole face of the TLC plate with the developer thus washing away the drops added and not letting the solvent “crawl” up the plate. This would make results invalid and in fact not show results at all. Also the picture of the TLC plate in Part C may have not been clear enough and or the dots were not dark enough for the ImageJ to produce valid results.

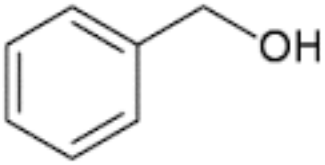
Questions

1. Polar solvents make the molecules of the compounds move faster and higher up the TLC plate. In part B, when the solvent was just Ethyl Acetate, the molecules of the compound had a much bigger affinity to the solvent than the silica gel, and this is why the dots appeared right below the solvent line. The more polar the solvent the more the molecule moves up the plate compared to less polar solvents that have less of an electromagnetic pull and thus the molecules move slower and less higher up the plate because the molecules are more attracted to the silica gel. Larger Rf values happen with polar solvents as opposed to less polar solvents.

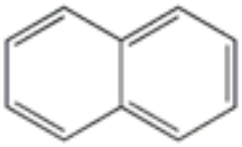
2.
a) From largest Rf value to smallest Rf value:
Benzyl acetate, benzaldehyde, benzyl alcohol
The more polar the compound the smaller the Rf value. The benzyl alcohol is the most polar because of its OH functional group on the end.

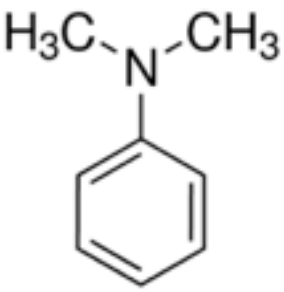


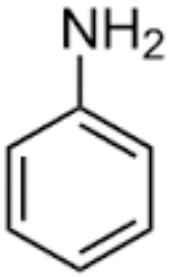




b) From largest Rf value to smallest Rf value:
Naphthalene, N,N-dimethylaniline , Aniline
The amine group attached to the Aniline makes it more polar than the other two compounds.







Conclusion

Through thin layer chromatography, in Part A it was found that the unknown compound #57 was made up of benzophenone and that the unknown compound YY in Part C is composed of 2.5: 1 M-bromonitrobenzene : P-bromonitrobenzene. As well as in Part B using Ethyl Acetate as a solvent proved that polar solvents make molecules move faster and higher up the TLC plate because all three dots appeared in a straight line right below the solvent line.

CHM TA: Jan 19.16

Ryan Ivanovich
rivon099 @ uottawa.ca

Labs due @ beginning of next lab (2:30 pm)

Brief reports are encouraged
↳ hence point - form is good
↳ reports need to be typed
↳ drawings are scanned & attached
↳ or use ChemDraw to make drawings
↳ online submission (as single file ADF)

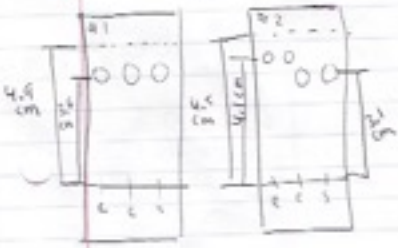
LAB # 1 Chromo

PART A

Unknown compound # 5 #
↳ white ~~powder~~ crystal / salt / Rubber smell
Ethyl Acetate : Hexanes 2:8
↳ smells like nail polish remover.

↳ a. benzophenone / clear / transparent liquid
↳ b. phenyl clear / transparent liquid
↳ outdoor house paint.

#1 TLC plate is benzophenone reference
#2 TLC plate is b. phenyl reference
both have sample of unknown compound + dichloromethane

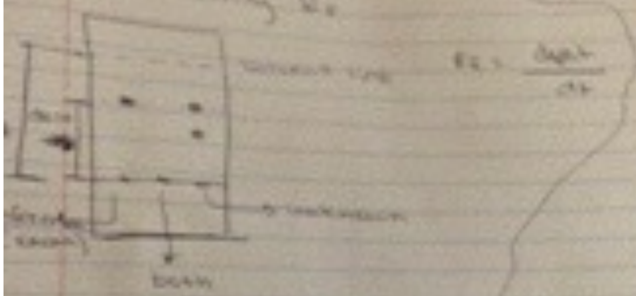


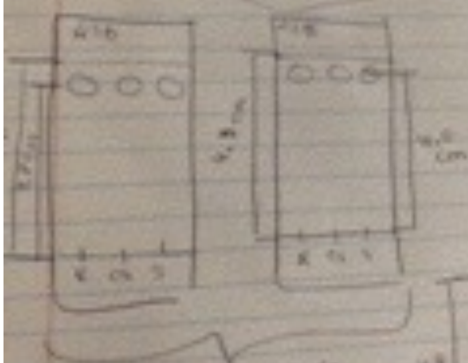
Our unknown is benzophenone because in the 1st TLC they are all at the same level (same Rf) that means co spot w/ two solvents both are benzophenone where as the #2 plate you can see the difference because the co-spot has two diff dots indicating two dif solvents

Part B

Solvent: Ethyl acetate (clear liquid / nail polish smell)

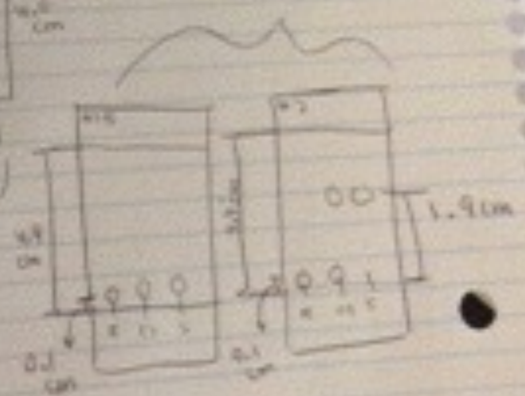
Calculating R_f





Ethyl acetate
Developer

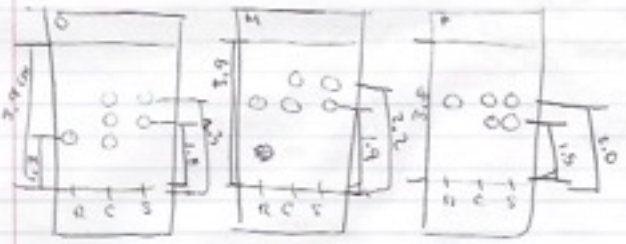
Hexane : clear liquid
smells like
wood polish



PART C

Unknown YY, clear / transparent liquid
↳ window cleaner

References:
O = transparent, yellow hue, liquid
M = transparent, clear, liquid
P = " " "



This shows that the unknown YY is composed of M and P. First of all you know that YY is composed of 2 molecules b/c of the 2 dots on the plate. b/c the two dots do not ~~match~~ line up w/ the ~~dot on the~~ ref. dot on the O plate that means YY is not composed of O. Yet M and P line up with either one of the YY dots — meaning that YY is composed of M + P.