Thin Layer Chromatography

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Section: Z04, locker 24

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Experiment 1: Thin Layer Chromatography

Protocol/Procedure:

- See laboratory manual for CHM1321, section for experiment #1: Thin Layer Chromatography, pages 1-6 (Venkateswaran, Sep. 2014).
- No modifications performed

Observations (Parts A, B, and C):

- All solutions remained clear
- No solutions bubbled or released gas
- Reference solutions volatile: strong fumes and smell from most compounds, notably that acetone and its trademark, dry and rank scent
- Fingerprints tainted certain results, so they had to be done again, since contaminated data isn't exactly valid. For future reference: strips must be handled as carefully as possible and minimize contact of silica gel with anything except graphite pencils and respective compounds

TLC drawings (annotated):

Figure 1: Part A- Using benzophenone and biphenyl to solve for an unknown

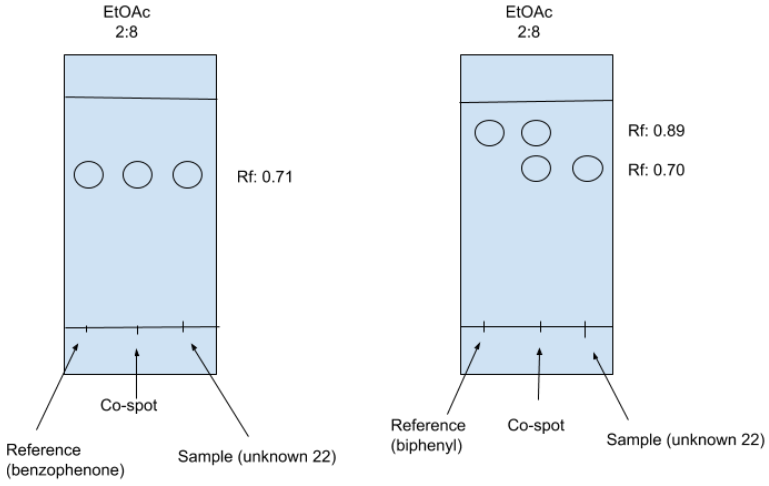


Figure 2: Part B- Effects of EtOAc

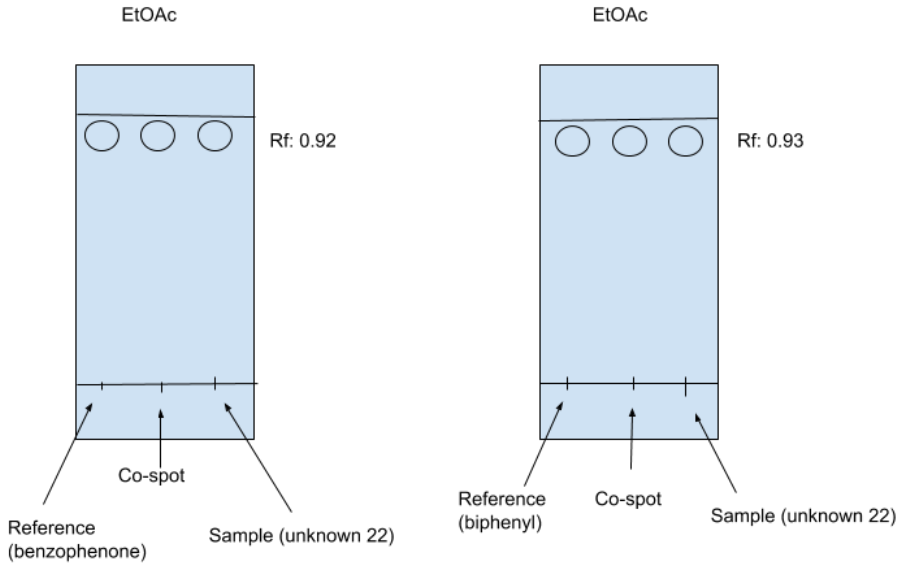


Figure 3: Part B- Effects of Hexanes

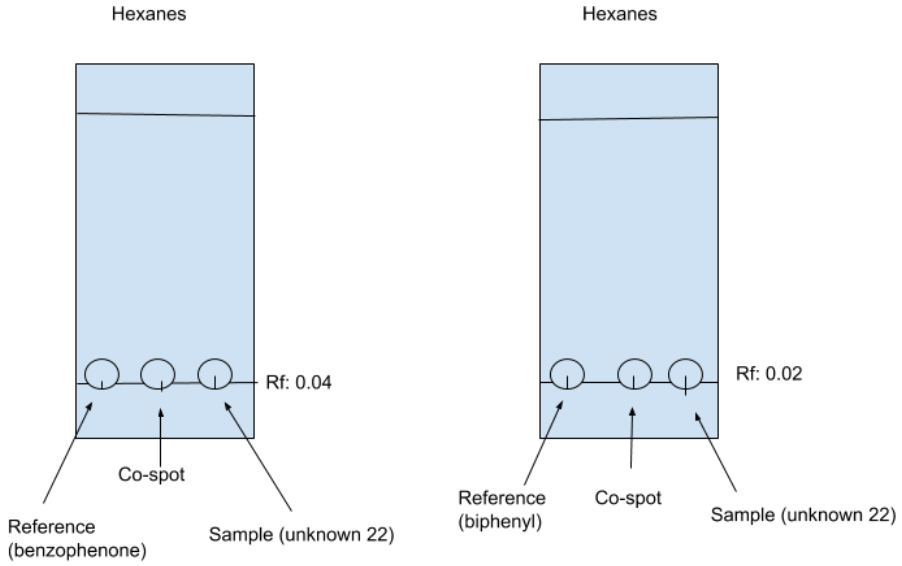
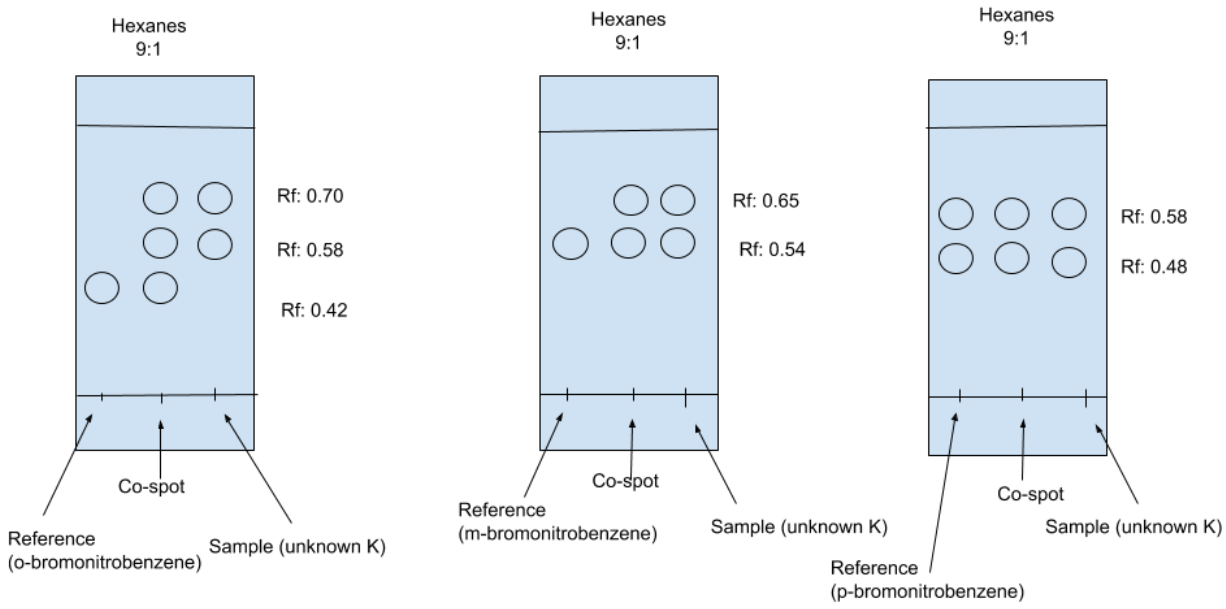


Figure 4: Part C- Identifying an unknown



Example R_f calculation:

Sample Calculations

R_f Calculations

$R_f = \frac{D1}{Ds}$

$R_f = \frac{32mm}{45mm}$

$R_f = 0.71$

*Taken from the R_f calculation for part A (very first calculation)

*Note: %mole of unknown and calibration for part C aren't required for this specific experiment.

Results:

Part A

Table 1: R_f values for reactions outlined in part A of the procedure.

	Ds (mm)	Reference (mm)	Co-spot (Reference) (mm)	Co-spot (Sample) (mm)	Sample (mm)	Rf
Trial 1 - Benzophenone	45	32	32	32	32	0.71
Trial 1 - Biphenyl	44	39	39	31	31	Reference: 0.89 Sample: 0.70

Part B:

Table 2: R_f values for reactions outlined in part B of the procedure.

	Ds (mm)	Reference (mm)	Co-spot (Reference) (mm)	Co- spot (Sample) (mm)	Sample (mm)	Rf
Ethyl acetate - Benzophenone	49	45	45	45	45	0.92
Ethyl acetate - Biphenyl	46	43	43	43	43	0.93

	Ds (mm)	Reference (mm)	Co-spot (Reference) (mm)	Co-spot (Sample) (mm)	Sample (mm)	Rf
Hexane -	45	2	2	2	2	0.04

Benzophenone						
Hexane - Biphenyl	45	1	1	1	1	0.02

Part C:

Table 3: R_f values for reactions outlined in part C of the procedure.

	Ds (mm)	Reference (mm)	Co-spot (Reference) (mm)	Co-spot (Sample) (mm)	Co-spot (Sample 2) (mm)	Sample (mm)	Sample 2 (mm)
o-bromonitrobenzene	50	21	21	35	29	35	29
m-bromonitrobenzene	46	25	25	30	25	30	25
p-bromonitrobenzene	48	29 23	29 23	28	23	28	23

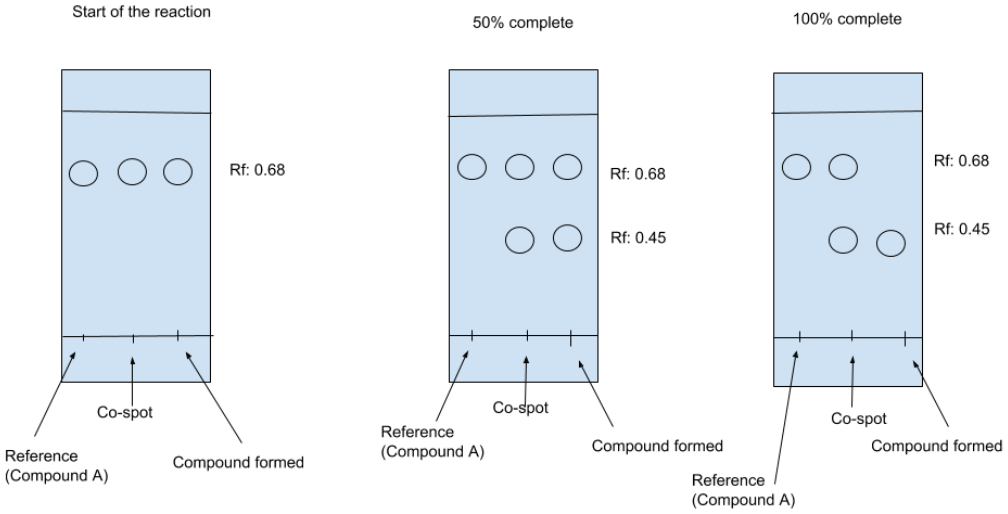
*Note: Two spots appeared under the reference point for the TLC with p-bromonitrobenzene

	Rf
o-bromonitrobenzene	Reference: 0.42 Sample: 0.70 Sample 2: 0.58
m-bromonitrobenzene	Reference: 0.54 Sample: 0.65 Sample 2: 0.54
p-bromonitrobenzene	Reference: 0.58 Reference 2: 0.48 Sample: 0.58 Sample 2: 0.48

Questions:

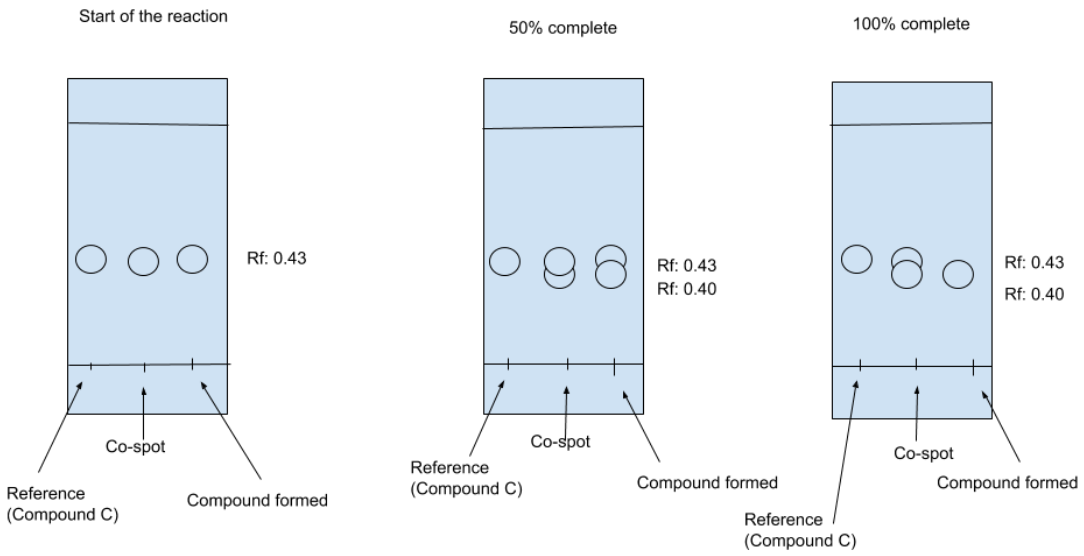
1. It is important to make the co-spot last to give the first compound enough time to evaporate from the plate.

2. By increasing the polarity of the solvent, the compounds move faster across the TLC. This is due to the disruption of the intermolecular forces between the silica gel and the compound.
3.



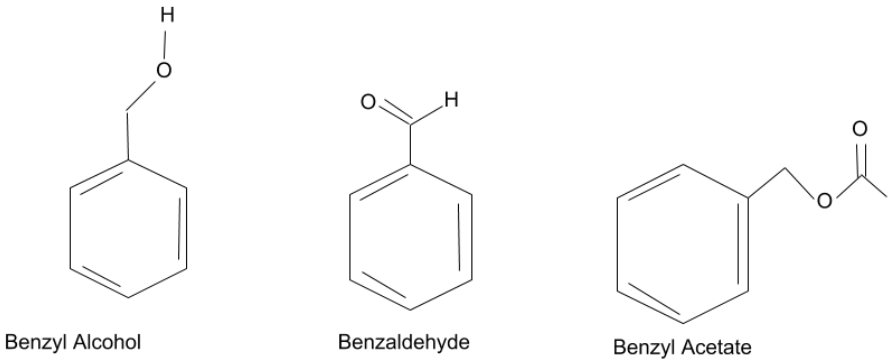
It is better to use a sample of compound A instead of compound B to follow the reaction because it is easier to see that the reaction is 100% when no spots are aligned with the reference.

4.

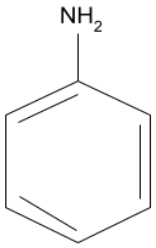


It is important to use a co-spot because the 2 compounds have close Rf values, it could be hard to tell if the spots are aligned. With a co-spot it is easier to align the spots of each compound.

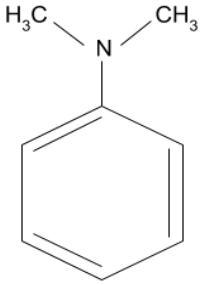
5.



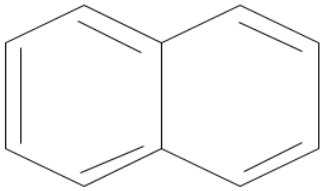
Most to least polar: Benzyl alcohol, Benzaldehyde, Benzyl Acetate
Benzyl alcohol is the most polar of these 3 compounds because of the hydrogen bond with the oxygen. Benzaldehyde is the second most polar of these compounds because it lacks a hydroxyl group making it less polar than benzyl alcohol but more polar than benzyl acetate because aldehydes are more polar than esters.



Aniline

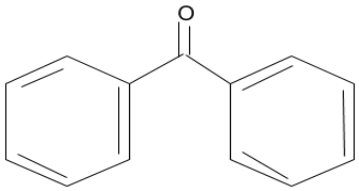


N,N-dimethylaniline

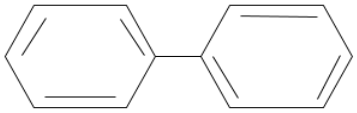


Naphthalene

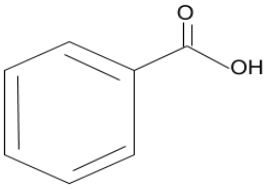
Most to least polar: Aniline, N,N-dimethylaniline, Naphthalene
Aniline is the most polar because the the difference in electronegativity between nitrogen and hydrogen is greater than the difference in electronegativity between nitrogen and carbon as present in N,N-dimethylaniline. Naphthalene is the least polar because it is non-polar since it only contains carbon-carbon bonds and carbon-hydrogen bonds.



Benzophenone



Biphenyl



Benzoic Acid

Most to least polar: Benzoic acid, Benzophenone, Biphenyl
The benzoic acid acid is the most polar because of the hydrogen bonds with the 2 oxygen atoms. Benzophenone is less polar than benzoic acid because there are no hydroxyl groups but more polar than biphenyl because is non-polar because it only contains carbon-carbon bonds and carbon-oxygen bonds.

Discussion:

Part A

- Unknown 22 had an average Rf value of 0.70

- Biphenyl had an average Rf of 0.89
- Benzophenone had an average Rf of 0.71
- Unknown substance 22 is more polar than biphenyl, as it progressed less along the solid phase.
- Unknown 22 is equally as polar than benzophenone, as it reached exactly as far as benzophenone on the solid phase
- This indicates that unknown 22 is benzophenone

Part B (ethyl acetate)

- Unknown 22 had the same Rf values as the reference substances (0.92 and 0.93)
- This would indicate that unknown 22 contains benzophenone. Pure ethyl acetate is a more polar solvent than the hexane mixtures and pure hexanes. Because biphenyl and benzophenone are non-polar, they can travel further in a polar solvent, which explains the higher R_f values.

Part B (hexanes)

- Unknown 22 had an Rf value of 0.04 for the benzophenone trial, and 0.02 for the biphenyl trial
- Benzophenone had an Rf value of 0.04. Biphenyl had an Rf value of 0.02
- Unknown is equally as polar as benzophenone, and more polar than biphenyl
- This indicates unknown 22 contains benzophenone, but not biphenyl

Part C

- The two spots for Unknown K had an average Rf value of 0.64 and 0.53
    - The more polar spot had Rf values of 0.70 and 0.65 in the trials for o and m-bromonitrobenzene, but trial p had an Rf value of 0.58.
- O-bromonitrobenzene had an Rf value of 0.42
- M-bromonitrobenzene had an Rf value of 0.54
- P-bromonitrobenzene had an Rf value of 0.58 and 0.48
- The more polar spot in unknown K has the same Rf value as m- bromonitrobenzene
- The less polar spot in unknown K has the same Rf value as p- bromonitrobenzene
- This indicates unknown K contains m-bromonitrobenzene and p-bromonitrobenzene, but not o-bromonitrobenzene

*A possible source of error, as aforementioned, would be the contamination of data via contact of silica gel with fingers and of other by products simultaneously in the lab. To avoid this, in the future, one must be more precise when handling the strips. This could affect the data by making it less accurate and providing us with an erroneous yield of the relative polarity of given compounds. Luckily, data lines up with theoretical expectations.

Conclusion:

Thin layer chromatography reactions were examined in this experiment successfully. In terms of identifying the unknowns; for part A and part B, it suggested that unknown #22 contained benzophenone, not biphenyl. For part C, unknown K was found to contain m-bromonitrobenzene and p-bromonitrobenzene, no o-bromonitrobenzene. Moreover, one can conclude that hexane is a way less polar solvent than Ethyl Acetate.

References:

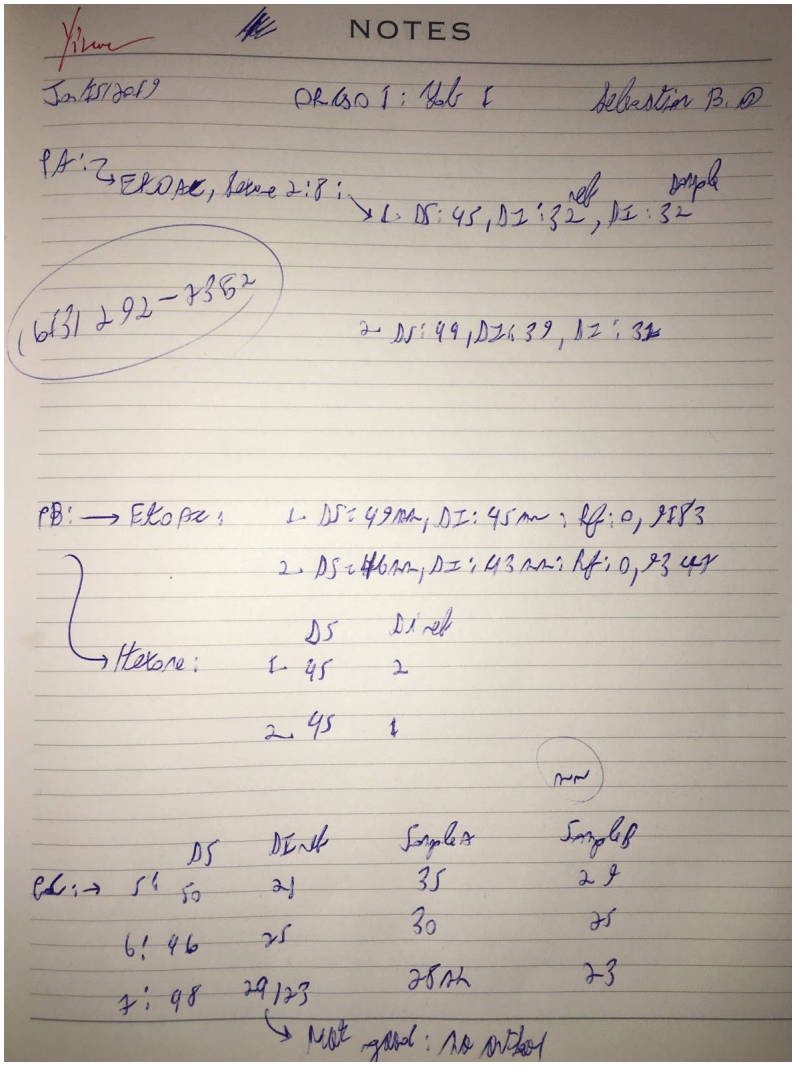
Clark, J. (2016). Thin Layer Chromatography. Retrieved from

https://www.chemguide.co.uk/analysis/chromatography/thinlayer.html

Olmsted, John, et al. *Chemistry*. John Wiley & Sons Canada, Ltd., 2016.

Venkateswaran, R. (2019) Thin Layer Chromatography.

Signed raw lab data, both parties:



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Experiment Part A

Unknown: 22

TLC	ds	d_1 ref	d_1 sample
1	45 mm	32mm	32mm
2	44 mm	39mm	31mm

Experiment Part B

TLC	ds	d_1 ref	d_1 cospot	d_1 sample	R_f
1	49 mm	45 mm			0.918367346
2	46 mm	43 mm			0.934782608
3	45 mm	2 mm			0.04444444
4	45 mm	1 mm			

Vivian

Part 3

unknown: K

TLC	ds	d_1 ref ~~spot~~	d_1 Sample A	d_1 Sample B
5	50 mm	21 mm	~~30 mm~~ 35 mm	~~25 mm~~ 29 mm
6	46 mm	25 mm	30 mm	25 mm
7	48 mm	29 mm 23 mm	28 mm	23 mm

Note: two point appeared under the reference spot of TLC 7 even though there should only be one

Reference pictures of TLCs:

Top row is A, Middle B (Hexane, EtOAc, left to right, respectively) and bottom is C.