Chem 1321

Experiment 1: Thin Layer Chromatography

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Introduction

- **Thin Layer Chromatography (TLC)**: A specialized type of adsorption chromatography that use a thin layer of adsorbent (stationary phase) attached to a plate (usually made of glass or aluminum) and the mobil phase passes over the plate and through the stationary phase.
- TLC is used for reaction monitoring, for sample purification, and to determine the purity of samples.
- More polar compounds are attracted more strongly to the silica than to the solvent, and move more solely up the TLC plate
- Less polar compound are less strongly attract to the silica and move more quickly up the TLC plate
- The distance that both polar and non-polar compounds moves can be increased by increasing the polarity of the solvent.
- **R_F value** is a measure of the polarity of each component (spot). Molecules that are **non-polar** will have **large R_F value**. **Polar** molecules appear at the bottom of the TLC plate and have **small R_F value**.

Procedure

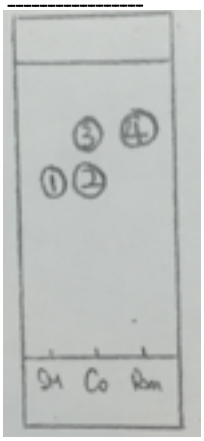
- Refer to the Lab Manual p.16-p.18

Observation

- When spot the solution on the TLC plate, the wet small spot appear on the baseline of the TLC plate but disappear quickly to it's original form.
- When put the TLC plate into the solvent of the jar, the TLC plate become wet and changes colour as the solvent rise up on the TLC plate
- Solution XX appear with yellow transparent colour with little excitant smell.

Result and Discussion

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



①Ref: Benzophenone

	d_1/cm	d_s/cm	R_F/cm
1	2.1	4.0	0.525
2	2.09	4.0	0.5225
3	3.2	4.0	0.8
4	3.3	4.0	0.825

②Ref:Biphenyl

	d_1/cm	d_s/cm	R_F/cm
1	3.1	3.9	0.795
2	3.1	3.9	0.795
3	3.1	3.9	0.795

Discussion

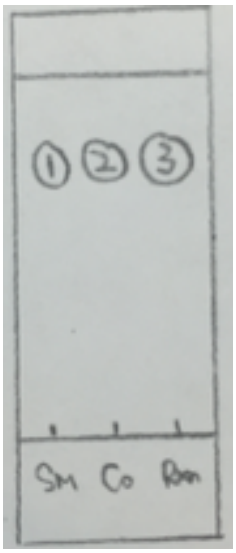
-For TLC plate ① with reference of Benzophenon has spot 3 and 4 locate higher than the spot 1 and 2. Also the R_F value of spot 3 and 4 is greater than the spot 1 and 2 which indicates that the sample solution is less polar than the reference solution in plate ①.

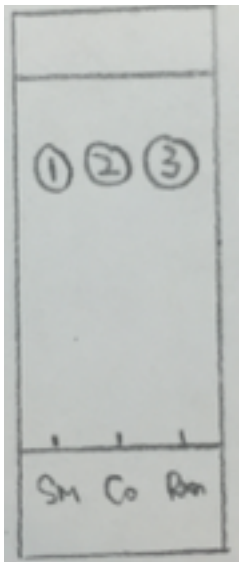
-For TLC plate ② with reference Biphenyl has all three spot locate at same level with R_F value also the same. This indicates that the sample solution are the same solution as reference solution.

- Compare both TLC plate ① and ②, since the mobile phase solution are the same and the R_F value for spot 3 and 4 in the plate ① is close to the R_F value of all 3 spots in plate ②, which indicates the unknown solution is also biphenyl and benzophenone solution is more polar than the biphenyl solution.

Part B: Effect of solvent on TLC

Solvent: Ethyl acetate (EtoAC)





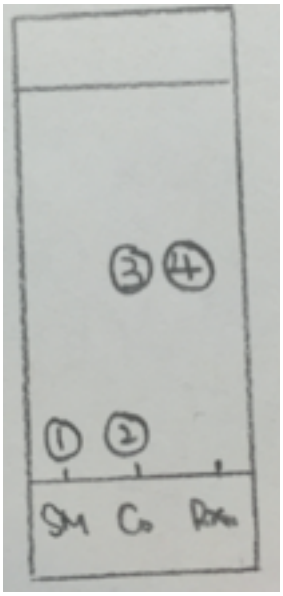
①Ref: Benzophenone

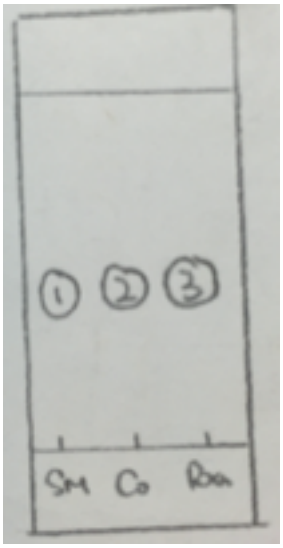
	d_1/cm	d_s/cm	R_F/cm
1	3.4	4.0	0.85
2	3.6	4.0	0.9
3	3.64	4.0	0.91

②Ref: Biphenyl

	d_1/cm	d_s/cm	R_F/cm
1	3.29	3.90	0.844
2	3.30	3.90	0.846
3	3.29	3.90	0.844

Solvent: Hexanes





③Ref: Benzophenone

	d_1/cm	d_s/cm	R_F/cm
1	0.12	4.0	0.03
2	0.05	4.0	0.0125
3	2,05	4.0	0.5125
4	2.10	4.0	0.525

④Ref: Biphenyl

	d_1/cm	d_s/cm	R_F/cm
1	1.90	3.91	0.486
2	1.91	3.91	0.488
3	2.20	3.91	0.512

Discussion

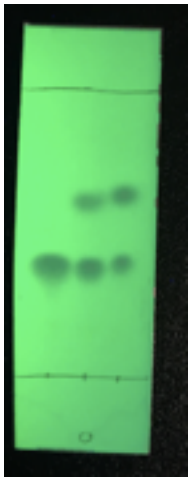
- For TLC plate ① with reference of Benzophenon has 3 spot locate nearly at the same level with R_F value of 0.85, 0.9, 0.91, spot 3 R_F value is still greater than the spot 1 R_F value, which indicates that the reference solution is more polar than the sample solution.
- For TLC plate ② with reference Biphenyl has all three spot locate at same level with R_F value also the same. This indicates that the sample solution are the same solution as reference solution.

- For TLC plate ③ with reference of Benzophenon has spot 3 and 4 locate higher than the spot 1 and 2. Also the R_F value of spot 3 and 4 is greater than the spot 1 and 2 which indicates that the sample solution is less polar than the reference solution in plate ①.
- For TLC plate ② with reference Biphenyl has all three spot locate at same level with R_F value also the same. This indicates that the sample solution are the same solution as reference solution.
- Compare both TLC plate ① and ③, since the R_F value of each spot in plate ① is greater than the R_F value of each spot in plate ③, since more polar solvent disrupts the intermolecular forces between the silica gel and compound and making compound move further alone the plate,which indicates that the solvent of solution Hexanes is less polar than the solvent of solution Ethyl acetate.
- Compare both TLC plate ② and ④, since the R_F value of each spot in plate ② is greater than the R_F value of each spot in plate ④, since more polar solvent disrupts the intermolecular forces between the silica gel and compound and making compound move further alone the plate which indicates that the solvent of solution Hexanes is less polar than the solvent of solution Ethyl acetate.

Part C: Ratio of Compounds (Unknown Solution XX)

- Point Order from left to right, down to top

Solvent Solution: Hexane: Ethyl Acetate (9:1)



①Ref: o-bromonitrobenzene

	d_1/cm	d_s/cm	R_F/cm
1	2.0	6.4	0.31
2	2.1	6.4	0.33
3	2.2	6.4	0.34
4.	3.6	6.4	0.56
5.	3.8	6.4	0.59

②Ref: m-bromonitrobenzene

	d_1/cm	d_s/cm	R_F/cm
1	1.9	6.0	0.32
2	1.9	6.0	0.32
3	2.8	6.0	0.47
4	2.7	6.0	0.45
5	3.4	6.0	0.57
6	3.3	6.0	0.55

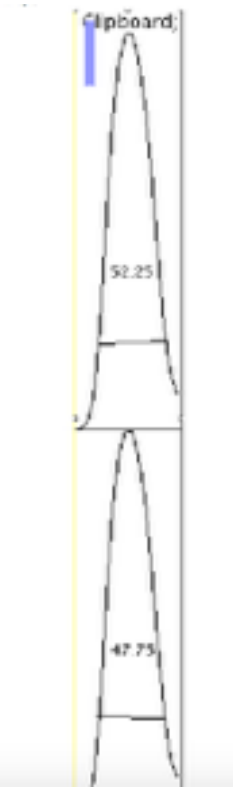
③Ref: p-bromonitrobenzene

	d_1/cm	d_s/cm	R_F/cm
1	2.1	7.0	0.30
2	2.3	7.0	0.33
3	4.1	7.0	0.59
4	3.9	7.0	0.56
5	4.0	7.0	0.57

Discussion

- Since in group ② with the reference of m-bromonitrobenzene does not have the spot locate at the same level as the reference, which indicates that the solution XX does not indicates the m-bromonitrobenzene but only the o-bromonitrobenzene and p-bromonitrobenzene.

- Ratio



	Area	Percent
1	5225.113	52.248
2	4775.406	47.752

% o-bromonitrobenzene=52.248%

% p-bromonitrobenzene=47.752%

Ratio: o-bromonitrobenzene=5.2
p-bromonitrobenzene 4.8

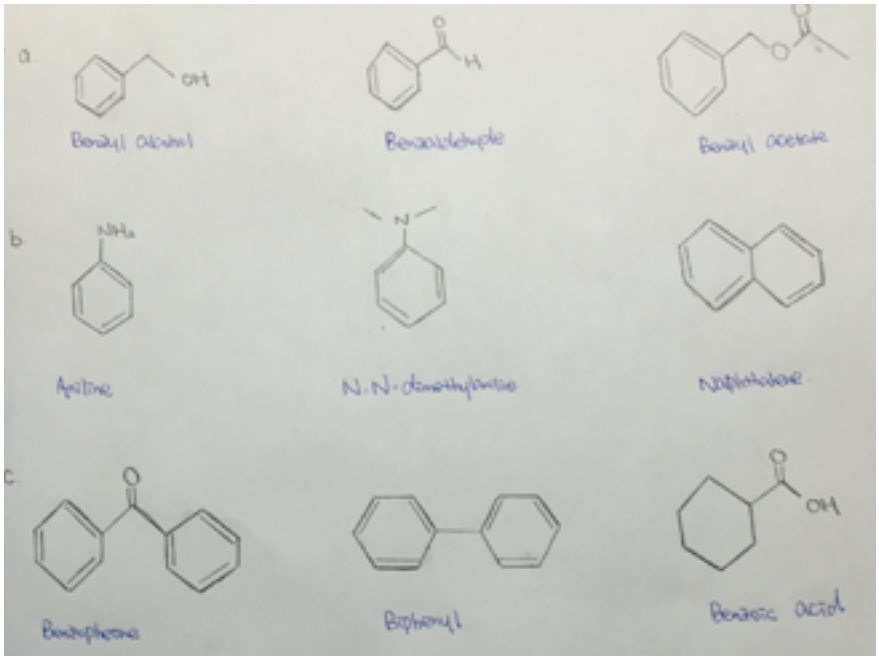
The XX solution contain o-bromonitrobenzene and p-bromonitrobenzene with the ratio 5.2: 4.8

Question

1. Increasing the polarity of the solvent will increase the distance for both polar and non-polar compounds move, since more polar solvent disrupts the intermolecular

forces between the silica gel and the compound, making the compound move further alone the plate.

2.



- In group a, the Benzyl belongs to the Alcohols group, Benzaldehyde belongs to the Aldehydes group and Benzyl acetate belongs to the Ethers group.
- In group b, all three compound belongs to the Aromatics
- In group c, the Benzophenone belongs to the Kentones group, Biphenyl belongs to the Aromatics group and Benzoic acid belongs to the Carboxylic Acids and Amids group.

- Compare all the compound in 3 groups, group a compounds are belongs to the group that are relatively have the slower mobility, which indicates that group a compounds react more with the TLC plate and more polar, which have the smallest R_F value on the silica gel.