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Lab 1: Thin Layer Chromatography

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Procedure

See laboratory manual pages 13-19.

Observations

Part A: The sample given is a white solid with a crystal-like structure, and is slightly translucent. It is sample 54. The sample was dissolved in dichloromethane, a clear liquid. Both references, biphenyl and benzophenone, are clear liquids. The solvent in the developing jar, a 2:8 mixture of ethyl acetate, is a clear liquid and smells strongly.

Part B: The same sample and references from part A were used, and the same characteristics were observed. The solvent in the developing jar in part B was ethyl acetate and later hexanes. Both were strong smelling, but the hexanes more so. Both were clear liquids.

Part C: The unknown, XX, was a translucent yellow liquid. The solvent system of 9:1 Hexanes: Ethyl Acetate was a strong smelling, clear, liquid. The controls, o-bromonitrobenzene, m-bromonitrobenzene, and p-bromonitrobenzene, are all clear liquids.

Rf Values

See appendix for TLCs.

Part A:

Solvent: 2:8 mixture of ethyl acetate

Reference	Rf of Refence	Rf of Sample 54
Benzophenone	0.60	0.78
Biphenyl	0.84	0.84

Part B:

Solvent: Ethyl acetate

Reference	Rf of Refence	Rf of Sample 54
Benzophenone	0.78	0.67
Biphenyl	0.84	0.80

Solvent: Hexanes

Reference	Rf of Refence	Rf of Sample 54
Benzophenone	0.16	0.55
Biphenyl	0.65	0.65

Part C:

Ortho:

Rf of reference (ortho)	Rf of Sample (xx)
0.80	0.45, 0.66

Meta:

Rf of reference (meta)	Rf of Sample (xx)
0.62	0.49, 0.70

P:

Rf of reference (p)	Rf of Sample (xx)
0.77	0.77, 0.50

Calculations

Rf Value

For Benzophenone in Part A, solvent is a 2:8 mixture of ethyl acetate.

Length from base line to top line: 4.5cm

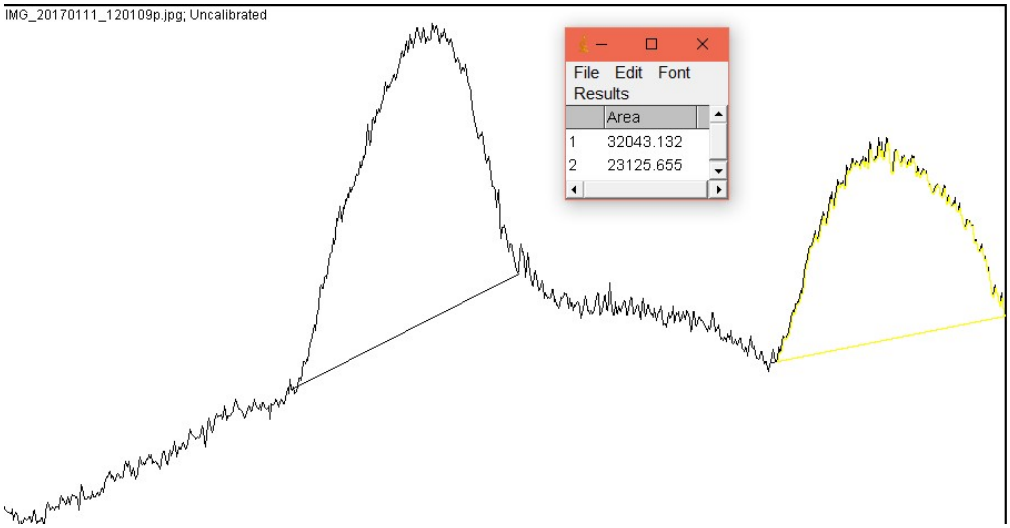
Length from base line to benzophenone spot: 2.7cm

$$Rf = \frac{2.7cm}{4.5cm}$$

$$Rf = 0.60$$

% Absorbance Calculation

Using ImageJ, pictured below.



$$\%\ absorbance\ P = \frac{(area\ of\ peak\ 1)}{(total\ area\ of\ peaks)} * 100\%$$

$$\%\ absorbance\ P = \frac{32043.132}{(32043.132 + 23125.655)} * 100\%$$

$$\%\ absorbance\ P = 58.1\%$$

$$\%\ absorbance\ M = 100\% - percent\ absorbance\ P$$

$$\%\ absorbance\ M = 100\% - 58.1\%$$

$$\%\ absorbance\ M = 41.9\%$$

% Mole Calculation

Using calibration curves.

$$y = 0.976x + 0.804$$

$$41.9 = 0.976x + 0.804$$

$$Mole\ percent\ of\ m = 42.1\%$$

$$Mole\ percent\ of\ o = 100\% - 42.1\%$$

$$Mole\ percent\ of\ o = 57.9\%$$

Discussion

In this lab, the were a total of 9 TLC plates developed. The first 6, from parts A and B, were using the same sample. The goal was to determine the composition of this sample as well as the effect of different solvents. The separation of the compounds on the TLC plates is dependent on the polarity of the sample, references, and solutes, and therefore a variety of TLC plates were tested to determine the effects of each.

Looking at the TLC plates from Parts A and B we can see a correlation between sample 54 and one of the references, biphenyl. The same references, biphenyl and benzophenone were used in parts, always along with sample 54. The solvent system was changed however. In all solvent systems, the Rf values of biphenyl and sample 54 are close. In Part A, both Rf values were 0.84. In part B, the first values of biphenyl and the sample were 0.84 and 0.80, respectively. The second values for part B were 0.65 for both the sample and the reference.

For Part C, ImageJ is used to calculate the percent composition. Looking at our Rf values, we can be confident that our unknown, XX, contains *p*-bromonitrobenzene, as their Rf values correspond. Looking at the TLCs, there are two dots for the sample, so we know sample XX must also contain something else. The Rf values of these dots are 0.45, 0.49, and 0.50. These do not directly correspond to either *o*-bromonitrobenzene or *m*-bromonitrobenzene, which have Rf values of 0.80 and 0.60, respectively. However, the values are much closer to that of *m*-bromonitrobenzene, so the calculations will be done assuming that is the other compound. Since only one trial was done with *m*-bromonitrobenzene, the confidence in that value is not high, and it could be in fact closer to that of XX. Upon calculation, it was found that the compound XX was 42.1% *m*-bromonitrobenzene and 57.9% *p*-bromonitrobenzene.

If this lab was conducted again, it could be improved upon by adding more trials to part C in particular. Because there was only one trial for each reference, there is not a high amount of confidence in these values. In addition, more practice with TLC strips should be accumulated before the lab to avoid contamination of samples. Some samples had additional dots where they should not have been, and though results could still be interpreted, had the dots been in a different location, the results could have been affected.

1. When the polarity of a solvent system is increased, the compound moves up higher in the silica gel. In our experiment, the spots on the TLC are higher when using ethyl acetate, which is more polar solvent. It gave an Rf value of 0.84 for biphenyl. When using hexanes, a non-polar solvent, the spots had an Rf value of 0.65.
2. In a set, compounds with a greater polarity will have the smallest Rf value, because the more polar the compound is, the more it will adhere to the polar silica gel. *All drawings are in the appendix.*
    a. In this set, the most polar was benzyl alcohol, and therefore will have the smallest Rf value. It is the most polar because it has an alcohol functional group, rather than an aldehyde or ester.
    b. In this set, the most polar was aniline, as it has an amine group with 2 hydrogens and a lone pair of electrons on the electronegative nitrogen, allowing for lots of hydrogen bonding sites. This also means it will have the lowest Rf value.

c. In this set, the most polar was benzoic acid, due to its carboxylic acid functional group, which is more polar than biphenyl, which is aromatic, or benzophenone, which is a ketone. This means in will have the lowest Rf value.

Appendix: TLC's

Part A: TLC 1

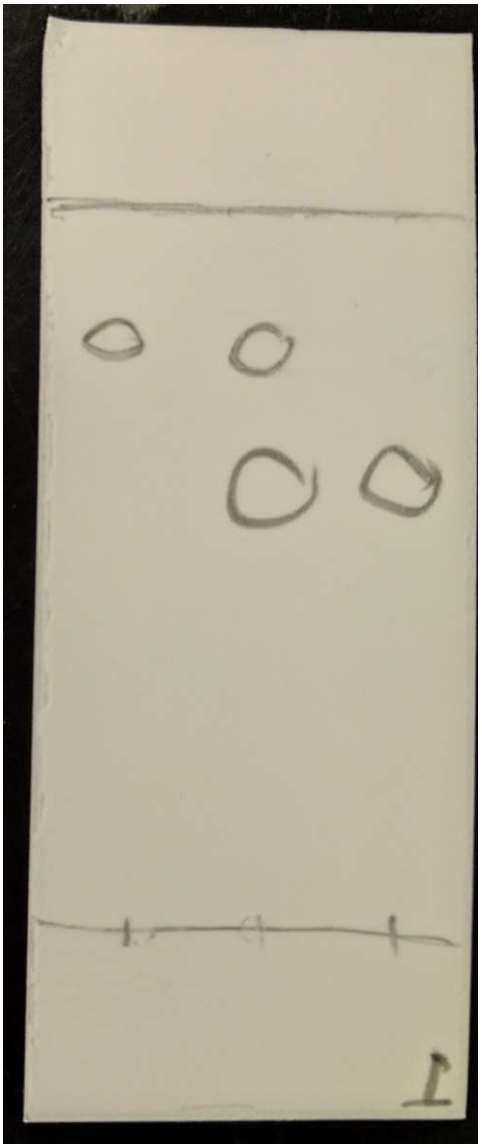
Solvent system: 2:8 mixture of ethyl acetate

Left column: Reference (biphenyl)

Middle Column: Reference and Sample

Right Column: Sample #54

Baseline is the lower line, solvent line is the upper line.



Part A: TLC 2

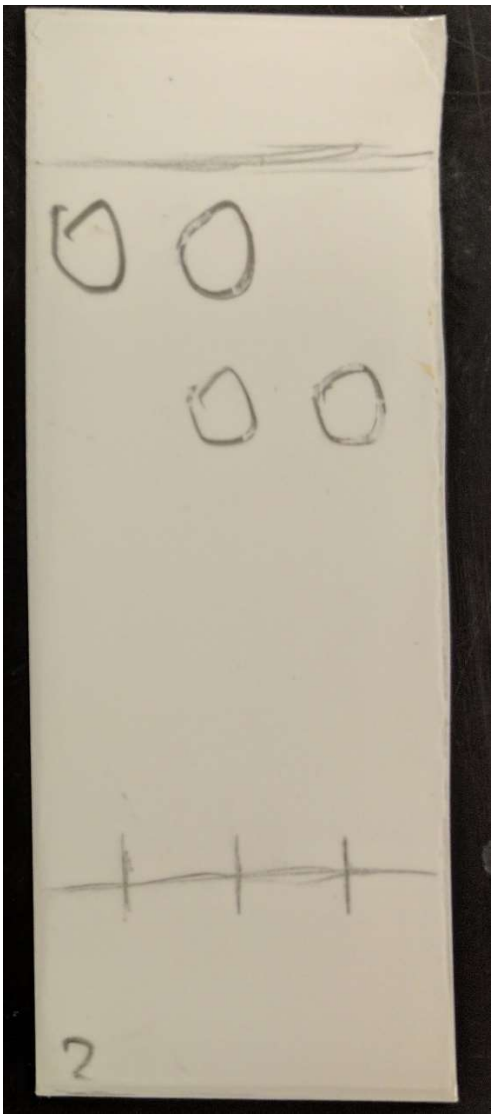
Solvent system: 2:8 mixture of ethyl acetate

Left column: Reference (benzophenone)

Middle Column: Reference and Sample

Right Column: Sample #54

Baseline is the lower line, solvent line is the upper line.



Part B: TLC 1

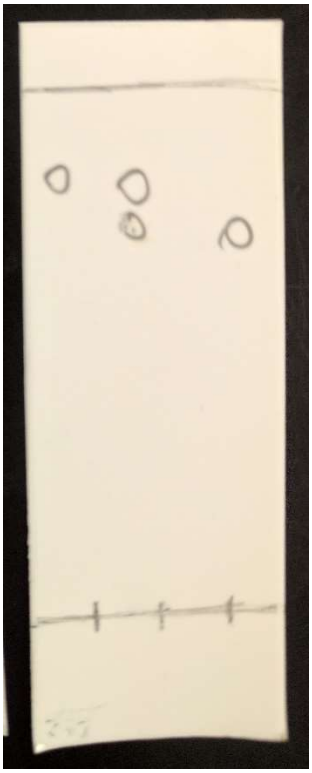
Solvent system: ethyl acetate

Left column: Reference (biphenyl)

Middle Column: Reference and Sample

Right Column: Sample #54

Baseline is the lower line, solvent line is the upper line.



Part B: TLC 2

Solvent system: ethyl acetate and later hexanes

Left column: Reference (benzophenone)

Middle Column: Reference and Sample

Right Column: Sample #54

Baseline is the lower line, solvent line is the upper line.

Part B: TLC 3

Solvent system: hexanes

Left column: Reference (biphenyl)

Middle Column: Reference and Sample

Right Column: Sample #54

Baseline is the lower line, solvent line is the upper line.

Part B: TLC 4

Solvent system: hexanes

Left column: Reference (biphenyl)

Middle Column: Reference and Sample

Right Column: Sample #54

Baseline is the lower line, solvent line is the upper line.

Part C: TLC 1

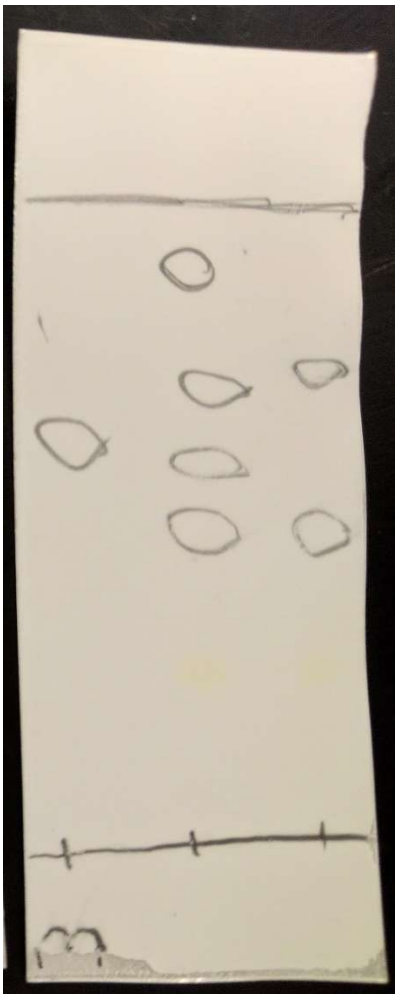
Solvent system: 9:1 Hexanes: Ethyl Acetate

Left column: Reference (m-bromonitrobenzene)

Middle Column: Reference and Sample

Right Column: Sample XX

Baseline is the lower line, solvent line is the upper line.



Part C: TLC 2

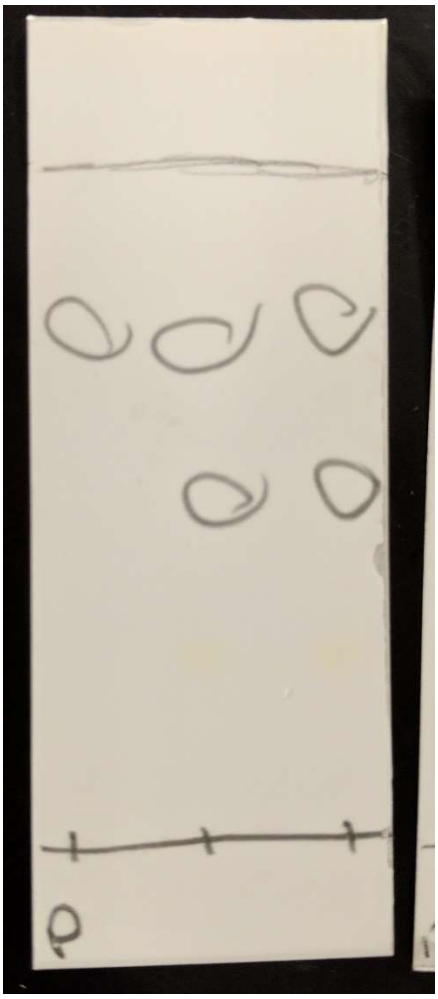
Solvent system: 9:1 Hexanes: Ethyl Acetate

Left column: Reference (p-bromonitrobenzene)

Middle Column: Reference and Sample

Right Column: Sample XX

Baseline is the lower line, solvent line is the upper line.



Part C: TLC 3

Solvent system: 9:1 Hexanes: Ethyl Acetate

Left column: Reference (o-bromonitrobenzene)

Middle Column: Reference and Sample

Right Column: Sample XX

Baseline is the lower line, solvent line is the upper line.