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Section: B01

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Experiment 1: Thin-Layer Chromatography

Date: 10th January 2017

Day and time: Tuesday, 2:30-5:30

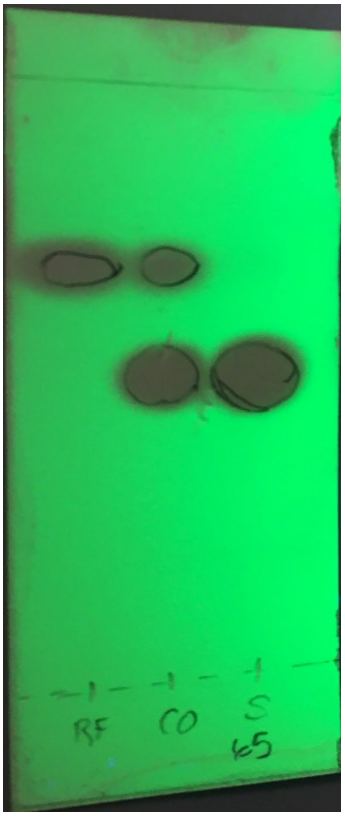
Protocol:

Please refer to the lab manual pages 13-19.
No modifications were made.

Observations:

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

TLC Sample #65



← Solvent line

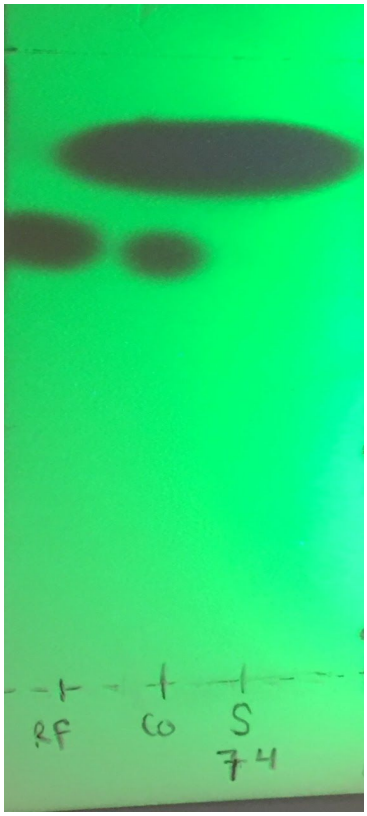
TLC of sample number 65 with biphenyl for reference. The solvent used for the sample was 2 mL of dichloromethane.
The solvent used for the TLC was 10 mL of a 2:8 mixture of ethyl acetate - EtOAc - and hexanes.

Rf value for biphenyl: 0.712
Rf value of sample 65: 0.539

← Baseline

↑ Biphenyl
↑ Both Biphenyl & 65
↑ sample 65

TLC Sample #74



← Solvent line

TLC of sample number 74 with benzophenone for reference. The solvent used for the sample was 2 mL of dichloromethane.
The solvent used for the TLC was 10 mL of a 2:8 mixture of ethyl acetate - EtOAc - and hexanes.

Rf value for benzophenone : 0.712
Rf value of sample 74: 0.846

← Baseline

↑ Benzo-phenone

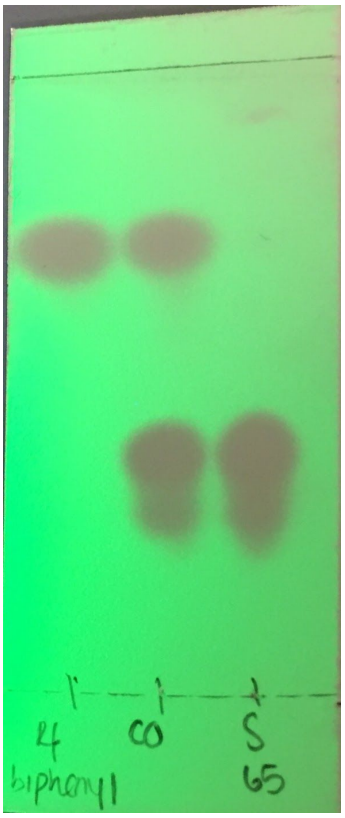
↑ both benzo-Phenone & 74

↑ sample 74

Part B: Effects of solvents on TLC

In Hexane:

TLC Sample #65



← Solvent line

TLC of sample number 65 with biphenyl for reference. The solvent used for the sample was 2 mL of dichloromethane.
The solvent used for the TLC was 10 mL of hexanes..

Rf value for biphenyl: 0.692
Rf value of sample 65: 0.346

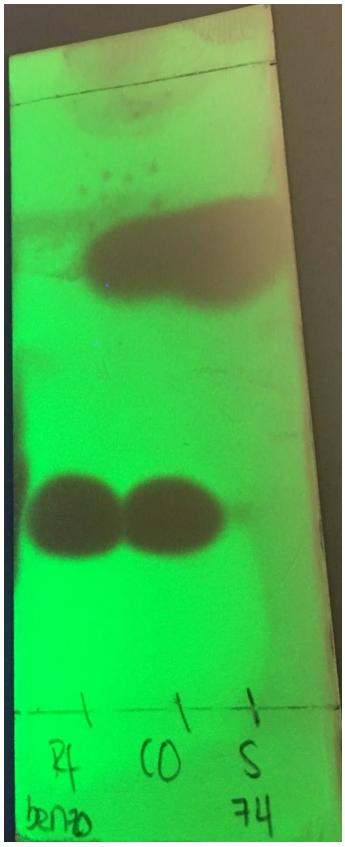
← Baseline

↑ Biphenyl

↑ Both Biphenyl & 65

↑ sample 65

TLC Sample #74



← Solvent line

TLC of sample number 74 with benzophenone for reference. The solvent used for the sample was 2 mL of dichloromethane.
The solvent used for the TLC was 10 mL of hexanes.

Rf value for benzophenone : 0.346
Rf value of sample 74: 0.692

← Baseline

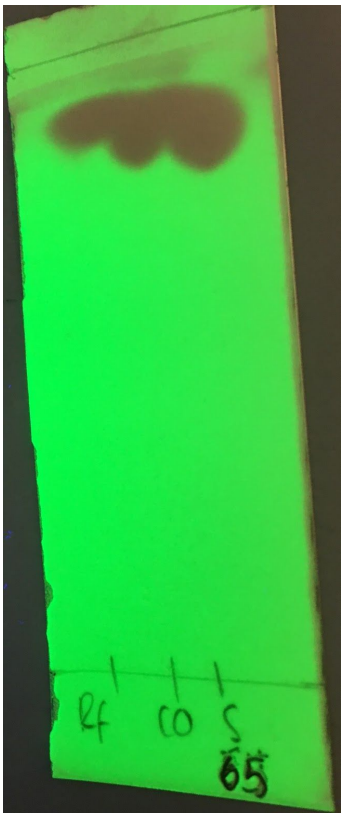
↑ Benzo-phenone

↑ both benzo-Phenone & 74

↑ sample 74

In Ethyl acetate:

TLC Sample #65



← Solvent line

TLC of sample number 65 with biphenyl for reference. The solvent used for the sample was 2 mL of dichloromethane.
The solvent used for the TLC was 10 mL of ethyl acetate.

Rf value for biphenyl: 0.788
Rf value of sample 65: 0.788

← Baseline

↑ Biphenyl

↑ Both
Biphenyl & 65

↑ sample 65

TLC Sample #74

← Solvent line

TLC of sample number 74 with benzophenone for reference. The solvent used for the sample was 2 mL of dichloromethane.
The solvent used for the TLC was 10 mL of ethyl acetate.

Rf value for benzophenone : 0.923
Rf value of sample 74: 0.923

← Baseline

↑ Benzo-phenone

↑ both benzo-Phenone & 74

↑ sample 74

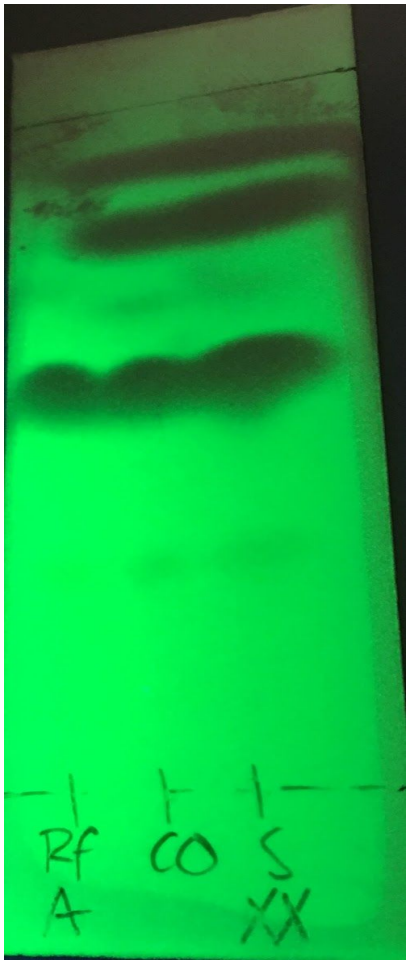
Part C: Ratio of compounds

TLC Sample XX #1

P3

P2

P1



Solution A

both Solution A and XX

sample XX

← Solvent line

← Baseline

TLC of sample XX with solution A for reference. Solution A is ortho-bromonitrobenzene. The solvent used for the sample
was 2 mL of dichloromethane.
The solvent used for the TLC was 10 mL a 9:1 solution of hexanes to ethyl acetate.

Rf for point 1 in solution A: 0.481
Rf for point 2 in solution A: 0.750
Rf for point 3 in solution A: 0.885
Rf for point 1 in sample XX: 0.481
Rf for point 1 in sample XX: 0.750
Rf for point 1 in sample XX: 0.885

TLC Sample XX #2

P2

P1

P3

← Solvent line

TLC of sample XX with solution B for reference. Solution B is meta-bromonitrobenzene. The solvent used for the sample was 2 mL of dichloromethane.

The solvent used for the TLC was 10 mL a 9:1 solution of hexanes to ethyl acetate.

Rf for point 1 in solution B: 0.635
Rf for point 2 in sample XX: 0.750
Rf for point 3 in sample XX: 0.500

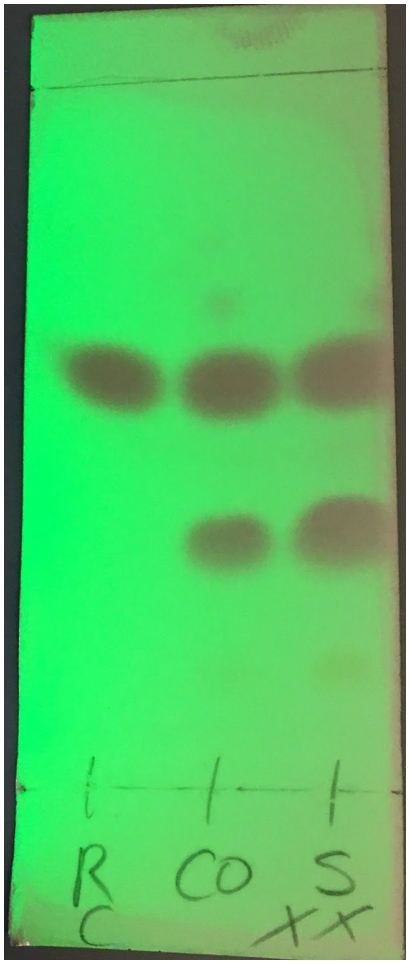
← Baseline

↑ Solution B

↑ both Solution B and XX

↑ sample XX

TLC Sample XX #3



← Solvent line

P1

P2

TLC of sample XX with solution C for reference. Solution C is para-bromonitrobenzene. The solvent used for the sample was 2 mL of dichloromethane.

The solvent used for the TLC was 10 mL a 9:1 solution of hexanes to ethyl acetate.

Rf for point 1 in solution C: 0.558
Rf for point 1 in sample XX: 0.558
Rf for point 2 in sample XX: 0.327

← Baseline

↑ Solution B

↑ both Solution B and XX

↑ sample XX

Calculations:

Calculating the Rf value:

Rf = *Distance from the baseline to the point*
Distance from the baseline to the solvent line

E.g. for sample 65 in solvent solution

$$Rf = \frac{2.8\ cm}{5.2\ cm}$$

$$Rf = 0.539$$

Calculating the percentage absorbance:

Absorbance of Para-Bromonitrobenzene = 34138
Absorbance of Ortho-Bromonitrobenzene = 30628
Percentage absorbance of Para-bromonitrobenzene = $(\frac{34138}{34138 + 30628})$ x 100% = 52.71%
Percentage absorbance of Ortho-bromonitrobenzene = $(\frac{30628}{34138 + 30628})$ x 100% = 47.29%

Calculating the mole percentage:

Y = %absorbance of para= 52.71%
Y = 1.0114x - 2.0208
Thus x = 54.121
%mol Para-Bromonitrobenzene = 54.121%
%mol ortho-Bromonitrobenzene = 46.489%

***Discussion**:*

Part A:

Sample 65 was a pure compound as it only had one point on the TLC. sample 65 is more polar than the solution of biphenyl as indicated by the Rf values of both solutions, the Rf value of the sample 65 was 0.539 which is lower than that of biphenyl which was 0.712. A lower Rf value indicates a more polar sample, as it decreases the interaction with the silica gel.

For sample #74, it still showed to be a pure sample as indicated by the one point that was shown in the TLC. sample #74 is less polar than the benzophenone as it had a higher Rf value of 0.846 in comparison to the benzophenone Rf value which is 0.712. Out of all 4 solutions, sample #75 was the least polar, followed by both biphenyl and benzophenone with equal polarities, and sample #65 was the most polar.

Part B:

In the solution of Hexanes, we can see that due to the fact that the hexane solution is not polar, it slowed down the samples and the reference solution quite significantly. This is probably due to the fact that the sample and the reference solutions are polar and hence using a non polar (or less polar) solvent system does not dissolve them as efficiently as a polar system. The Rf values for all the solutions decreased, in sample #65 the Rf value decreased from 0.539 to 0.346. In the biphenyl solution the Rf value dropped from 0.712 to 0.692, showing the smallest drop. In the benzophenone solution the Rf value dropped from 0.712 to 0.346 showing the largest drop, and the sample #74 solutio went from 0.846 to 0.692.

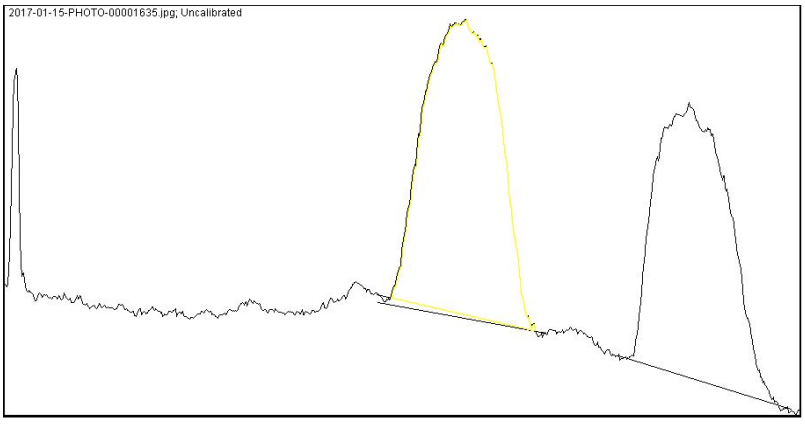
In the solution of Ethyl Acetate, we can see that due to the polarity of the solvent system, it accelerated the solubility of the solutions since they are also polar. All the Rf values increased significantly and in all cases the difference between the Rf values of the samples and the reference solutions diminished as due to how polar the solvent system was, the differences in polarity in the samples and the solutions was insignificant. In the first TLC both the sample #65 and the biphenyl solution had a Rf value of 0.788, while in the second TLC both the benzophenone solution and the sample #74 solution had the Rf value of 0.923.

Part C:

In part C we had an unknown sample labeled XX that was tested against 3 isomers, para-bromonitrobenzene, meta-bromonitrobenzene and ortho-bromonitrobenzene. The solvent system chosen was a 9:1 solution of hexanes : Ethyl acetate, this was used to provide a slightly polar solution that would not affect the Rf values of the various isomers significantly.

Sample XX showed to have 2 isomers as shown in having 2 points in the TLC. After comparing the Rf values of the points in sample XX against the Rf values of the various isomers, two Rf values matched up, 0.558 with Para-bromonitrobenzene, and point 0.481 with ortho-bromonitrobenzene. This led us to believe that the sample XX is a solution of Ortho-Para-bromonitrobenzene.

Statistical analysis was carried out to determine the mol percentage of the components of the isomer XX, we can see in the graph below the peaks representing the points in the TLC, with the areas in the enclosed peak representing the percentages of absorbance of both isomers. The peak to the right represents the ortho-bromonitrobenzene while the peak to the left represents the absorbance of the para-bromonitrobenzene. Which with further comparison against another graph allowed us to calculate the %mol composition of each isomer in the sample XX.



	Area
1	30627.735
2	34138.421

Errors:

Various errors were done in the experiment for example, human error was carried out while measuring the sample volume of the solvents, or during weighing the masses of the samples in part A and B. Further Human error could be carried out while measuring the distance from the baseline to the solvent line.

Other errors could include inaccurate ratios of the solvent systems due to difficulties stopping the pumps, transferring organic material to other parts of the TLC while touching it. Also some of the silica on the TLC paper was rubbed of with the pencil. Finally, the fact that there were no repeats to the TLC's producing less reliable Rf values.

Possible amelioration:

Possible ameliorations that could be made to improve the experiment procedure could be to increase the time given in order to allow more trials to be made, with more repeats the Rf values become more reliable. Moreover with increased time, it allows us to measure the quantities more accurately. In order to avoid issues with the proportions in the solvent system, using a pipette to transfer the chemicals is much more accurate and allows for less uncertainty. Another improvement could be to measure the Rf values 3 times and average the results, and to take better quality pictures for the analysis. Moreover using gloves could avoid cross contamination or transferring of organic material onto the TC, and using a more dull pencil could minimise the amount of silica rubbed off.

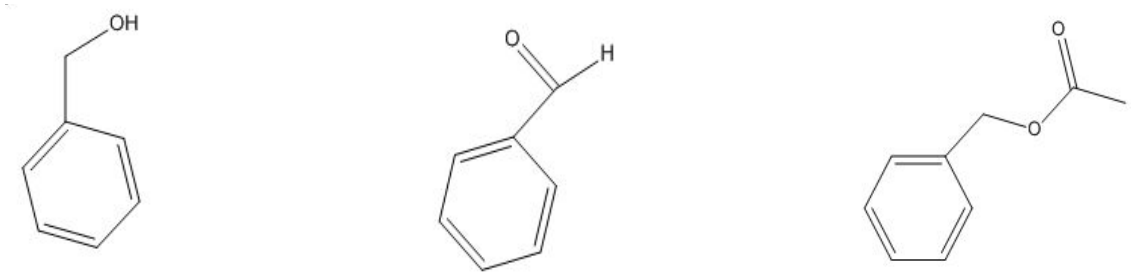
Questions:

1- How does increasing the polarity of the solvent system affect the results of a TLC?

Due to the fact that the polarity of the solvent system determines how far the substance being measured moves up a TLC, it has a huge impact on the Rf value. With an increase in polarity, there are more interactions between the substance being measured and the SiO in the silica gel in the TLC, hence slowing it down, and decreasing the Rf value. A non polar substance will have less interactions with the silica gel and hence move higher up, obtaining a higher Rf value. In conclusion, increasing the polarity of the solvent system decreases the Rf value, and vice versa.

2- In the following sets of compounds, which would have the smallest R_F on silica gel?

a. Benzyl alcohol: benzaldehyde: benzyl acetate:

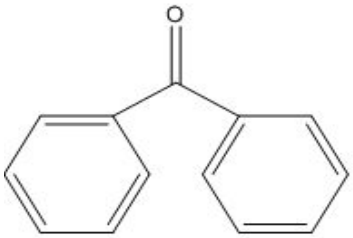


Benzyl alcohol will have the smallest Rf.

b. Aniline, naphthalene N,N-dimethylaniline,

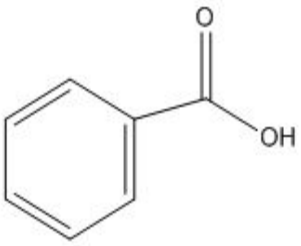
Aniline will have the smallest Rf.

c. Benzophenone



Biphenyl

Benzoic acid



Benzoic acid will have the smallest Rf.

Raw Data:

TLC of C as reference & sample XX

C : P_1 = 2.9cm

XX : P_1 = 1.7cm

P_2 = 2.9cm

TLC of B as reference & sample XX

B : P_1 = 3.3cm

XX : P_1 = 2.6cm

P_2 = 3.9cm

TLC of A as reference & sample XX

A : P_1 = 2.5 cm

P_2 = 3.9 cm

P_3 = 4.6 cm

XX : P_1 = 2.5 cm

P_2 = 3.9 cm

P_3 = 4.6 cm

10th January 2017

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