

Procedure:

Part A:

1. Added approximately 10 mL of 2:8 mixture of ethyl acetate (EtOAc) and hexanes to a developing jar and covered with a lid.
2. Prepared two TLC (Thin Layer Chromatography) as depicted in **Figure 1**. (appendix).
3. Obtained 10 mg of unknown sample #84 from demonstrator and dissolved the substance in a test-tube containing 2 mL of dichloromethane.
4. Using a capillary tube, spotted small dots of the dissolved sample solution onto the sample lanes and co-spot lanes of both prepared TLC plates.
5. 2 mL of benzophenone was added to a clean test tube. Small spots of the benzophenone reference solution was spotted onto the reference lane and co-spot lane of the first prepared TLC plate.
6. 2 mL of biphenyl was added to a clean test tube. Small spots of the biphenyl reference solution was spotted onto the reference lane and co-spot lane of the second prepared TLC plate..
7. Both TLC plates were then carefully placed into the developing jar containing ethyl acetate (EtOAc); ensuring that the silica layer was facing up and the pencil line was situated above the solvent level. The cover of the lid was then placed back on the developing jar, allowing the solvent to elute (approximately 3-5 minutes) until the solvent front was 1 cm from the top of the TLC plate.
8. Both TLC plates were then removed from the developing jar and the solvent finish line was marked lightly with a pencil. Afterwards the plates were placed aside to allow the solvent to evaporate from the plate.
9. Each TLC plate was then placed in a box containing a UV light, in which the visible spots were then gently circled using a pencil.

Part B:

1. Added approximately 10 mL of ethyl acetate (EtOAc) into a clean developing jar and covered with a lid.
2. Steps 2-9 from part A of the lab was repeated.
3. Added approximately 10 mL of ethyl hexanes into a clean developing jar and covered with a lid.
4. Steps 2-9 from part A of the lab was repeated.

Part C:

1. Demonstrator assigned an unknown substance labeled ZZ, in which we pipetted 2 mL of the unknown substance into a 50 mL beaker.

2. Three TLC plates were prepared as shown in **Figure 1.**, with each of the TLC plates acting as a reference compound: Octa-Bromonitrobenzen, Meta-Bromonitrobenze, and Petra-Bromonitrobenze.
3. Steps 2-8 from part A of the lab was repeated.
4. Pictures of each of the 3 TLC plates were taken under the UV light.

Please note that due to certain circumstances, the data collected for **Part C of the lab was provided by another group, permission was given by our T.A Tom Lacelle to do so**

Discussion:

Part A: The purpose of this portion of the lab was to use comparative measures between the reference solutions of Biphenyl and Benzophenone to identify the components of the unknown mixture of sample #84 using TLC. Each of the TLC plates were placed in a developing jar containing a 2:8 mixture of ethyl Acetate (EtOAc). Once the TLC plates were developed it was found that the unknown sample #84 yielded a near identical Rf value as the reference solution of Biphenyl, which would also indicate a similar polarity between the two. Based on these observations it can be deduced that the unknown sample #84 contained Biphenyl. When unknown substance #84 was tested using Benzophenone as the reference solution, the Rf values did not match, it indicated that Benzophenone had a higher Rf value than that of the unknown solution, indicating that Benzophenone was not found in the unknown sample #84 composition.

Part B: The purpose of this portion of the lab, was to determine the effects of varying solvents on TLC. Four different TLC plates were developed for this portion of this experiment: Two TLC plates containing the unknown sample #84 and Biphenyl as the reference solution were developed using the solvents Ethyl Acetate and Hexanes (one in each developing jar), and the other two TLC plates were completed while using Benzophenone as the reference solution. The results obtained showed that the Rf values of TLC that were developed in the Hexanes solvent, were lower of that of the TLC plates developed in the Ethyl Acetate solvent. This can be attributed to the concept of polarity difference of solvents, in which a less polar solvent can not as effectively disrupt the intermolecular forces between the silica gel and the compound, in comparison to a more polar solvent, thus the compound does not travel as far along the TLC plate. This is the case in which a Hexanes solvent is less polar than the Ethyl acetate solvent.

Part C: The purpose of this portion of the lab, was to determine ratio and percent composition of the mixture of compounds in the unknown ZZ.

Questions:

1. How does increasing the polarity of the solvent system affect the results of a TLC?
 1. By Increasing the polarity of the solvent system, the intermolecular forces between the silica gel and the compound are disrupted, thus allowing the compound to move further along the TLC plate.
2. In the following sets of compounds, which would have the smallest Rf on Silica gel? Use a sentence to explain your reasoning. Draw line structures of each Compound.

- A)** A higher polarity of a compound equates to a higher attraction to the silica gel, therefore move less further along the plate, resulting in a smaller Rf value. Due to the fact that Benzyl Alcohol is the compound that is most polar, Benzyl Alcohol is most likely to have the smallest Rf value.
- B)** A higher polarity of a compound equates to a higher attraction to the silica gel, therefore move less further along the plate, resulting in a smaller Rf value. Due to the fact that Aniline is the compound that is most polar, Aniline is most likely to have the smallest Rf value.
- C)** A higher polarity of a compound equates to a higher attraction to the silica gel, therefore move less further along the plate, resulting in a smaller Rf value. Due to the fact that benzoic acid is the compound that is most polar, benzoic acid is most likely to have the smallest Rf value.