

Thin Layer Chromatography

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Procedure:

Part A

1. Collect all equipment and materials needed to complete this section of the lab. These include: TLC plates, developing jars, unknown compound #10, dichloromethane, benzophenone and 2:8 ethyl acetate and hexane.
2. In the developing jar, place approximately 10 mL of 2:8 ethyl acetate and hexane.
3. Draw, in pencil, the starting line approximately 1 cm from the bottom of both TLC plates, as well as three evenly spaced tick marks on this line. One for the reference, co-spot and sample.
4. Dissolve roughly 10 mg of the unknown sample in a test tube with dichloromethane.
5. Using the capillary, spot one to two drops of the unknown onto both the plates in the sample lane and the co-spot lane.
6. Fill a separate test tube with benzophenone, and using a clean capillary spot the benzophenone in the co-spot and the reference lane of one TLC plate.
7. Fill a different test tube with biphenyl and spot this onto the co-spot and reference lane of the other TLC plate.
8. Place both TLC plates into the developing jar, ensuring that the silica layer is facing the inside of the jar.
9. Cover the jar and allow to sit for approximately 3-5 minutes, or until the solvent is about 1 cm from the top of the TLC plate.
10. Take the plates out of the developing jar and mark the finishing line with a pencil, then allow the plate to evaporate. This should take approximately 30 seconds.
11. Once the TLC plates are evaporated, place them under a UV light. Once under the light, circle the spots and measure from the center of each dot. using these values find the R_f values and identify the compounds that are in the unknown mixture.

Part B

1. Empty the content of the developing jar from Part A into the waste solvent bin and fill the jar with 10 mL of ethyl acetate.
2. Prepare two TLC plates. Refer to Steps 3-7 in Part A.
3. Place the plates into the developing jar, using ethyl acetate as the eluant, rather than 2:8 ethyl acetate and hexane.
4. Once the plates are developed, draw the finish line and allow them to evaporate. Place them under the UV light, marking the spots and drawing replicas of the plates.
5. Empty the content of the developing jar into the waste solvent bin and place 10 mL of hexane to use as the eluant.
6. Prepare two new TLC plates using the procedure from Steps 3-7 in Part A.

7. Place the plates into the developing jar with hexane and allow them to develop. When finished, draw the finishing line and allow them to evaporate.
8. Place them under the UV light and mark the spots. Then draw a replica.

Part C

1. Obtain a vial of an unknown mixture of compounds (for this experiment YY was used).
2. Prepare TLC plates as before, using o-bromobenzene, m-bromobenzene and p-bromobenzene as the reference compounds.
3. Place the plates under the UV light and take a picture of each TLC plate under the UV light before marking the spots.
4. Clean up work station.

Observations:

- TLC plates ended up 'messy' in Part B, as all the lanes had mixed together
- When two plates were in the same developing jar, one always seemed to develop faster than the other
- Ended up using two developing jars rather than using one. This made the experiment progress faster.

Results:

See attached page for replicas of the TLC plates

Part A

Part B

Part C

Questions:

1. The more polar a solvent is, the greater the R_f will be. This means that the spotted compounds will move higher up on the TLC plate at an increased rate.
2. a) Benzyl acetate will have the smallest R_f value because it is the largest molecule and will take longer to travel than the other substances.
b) *N,N*-diethylaniline will have the smallest R_f because it is a large molecule and polar. A more polar substance will take longer to travel through the silica gel on the TLC plate.
c) Benzophenone will have the smallest R_f because it is the largest of the three molecules and the most polar.

Discussion:

The first time doing Thin-Layer Chromatography is very trial and error based. If you spot too much or too little the TLC plates may not show a strong representation of what you are trying to achieve. For Part A and B there was too much spotting, even though only two drops of each were used, but the end result was very messy and hard to read.