Chem 224 Exp. #3 CER #707 TLC: Thin Layer Chromatography FS2017

Preparation of Developing Chambers

Obtain six labeled plastic beakers and watch glasses as developing tanks for the different solvents that will be used: **hexane, toluene, CH2Cl2, ethyl acetate, acetone and methanol.**

Two students share a set of beakers, but **each student should develop their own set of TLC plates**.

Line the inside of the beaker with a piece of large filter paper, then add about 10 ml or ¼" depth of each solvent to the appropriate beaker . Slosh solvent on the paper so that the vapors can saturate the air inside the beaker. Cover the beakers with a large watch glass, then prepare the TLC plates. Keep these beakers covered as much as possible since they stink and are bad for you.

TLC of Reference Compounds and Unknown Mixture

TLC strips containing spots of each of the three compounds will be tested in each of the six solvents to determine the best solvent for separation. Obtain six of the wide strips from the desiccator. Do not touch the surface of the strips with your fingers, as this will adversely affect the movement of the spots. Using a dull #2 pencil, not a pen, lightly draw a line ~1/2" from one end of each strip on the "chalky side". Label the top of each strip with one of the six solvents to be used.

Using a separate capillary for each compound, apply spots of the known solutions of biphenyl, benzophenone and benzhydrol to the line on each of the six strips. Try to keep the spots as small as possible. You should also label the top of each strip as to the identity of the spots and keep them in the same order for all six strips to avoid confusion. Several applications of each compound may be needed to get an adequate concentration of the spot. The hair dryer from your common drawer may be used to speed evaporation of the solvent between applications. I would also suggest that you check the strips under the UV (blacklight) viewing box prior to development to ensure that you have applied enough material so that the three spots are visible.
Caution: Do not look directly at the UV source lamp

Be sure that the level of the solvent in the beaker is below the line containing the spots (ie less than ½"). Place the strips in the appropriate solvent beaker, cover and allow to develop to within ~1/2" of the top. The amount of time required will vary with the solvent, and you should record the approximate time required for each. Remove the strips and mark the location of the solvent front lightly with a pencil. Use a hair dryer under your hood to evaporate the solvent, if necessary. Outline the spots on each strip lightly with a pencil while viewing under the blacklight. If the plate has a dark appearance under UV, the solvent has not been completely evaporated. Toluene is frequently found to do this as it has a higher BP than the other solvents.

Calculate the Rf for each compound for the two best solvent systems. Also calculate the approximate number of theoretical plates, N, by measuring the width of the spots, $N=16(d/w)^2$.

Obtain an unknown sample. Record your unknown# and run your unknown using the best solvent, which is the one that gives the widest separation of the known compounds. Determine which two compounds are present in your unknown by matching Rf values with the standards in the same solvent. If there is any uncertainty as to their identity, run a spot of the possible knowns alongside the unknown on a new plate.

Attach your TLC strips to the yellow pages of your lab book by covering them completely with 2" wide clear tape. Tabulate your results for Rf and N for the two best solvents. Be sure to record your unknown number and your identification of the compounds in your unknown. Comment on the outcome.