

Experiment: Column Chromatography

Experiment Date to be Performed: 3/1/2016

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Purpose: To separate two known compounds (ferrocene and fluoronone) using silica gel column chromatography after determining the appropriate separation conditions with TLC.

Procedure Outline:

- Using previously learned TLC, develop conditions (only available solvents are hexanes and ethyl acetate) to separate the two compounds to an R_f difference of at least 0.5
- Add small piece of cotton to column, force into neck with wire.
- Clamp column, add small plug of sand
- Fill column 2/3 full with solvent determined with TLC. Let sand settle. Tap column to level sand.
- Slowly add silica gel to column, let settle with each addition.
- Drain solvent as appropriate to keep column from overflowing
- DO NOT LET GO DRY!!! Add solvent as necessary
- Keep adding silica until column is 2/3 full of silica.
- DO NOT LET GO DRY!
- Tap column with rubber stopper to settle silica further and to level it.
- Drain solvent until just above silica gel
- Dissolve sample (1:1 by mass of the two cmpds) in a minimal amount of solvent.
- Add this to silica surface without disturbing level of silica
- Drain to level of silica
- Add minimal solvent to sample, add to silica and repeat above. Without disturbing level of silica
- Add small amount of fresh solvent to column and drain again. DO NOT LET GO DRY! Repeat.
- Add solvent, carefully add a layer of sand (protects silica from un-leveling), drain.
- Repeat until first compound elutes (check via TLC for presence).
- When first cmpd finishes eluting, switch to 100%EtOAc to push second cmpd through.
- DO NOT EVER LET COLUMN GO DRY!
- Remove solvent via stream of air
- Let dry overnight and obtain mass recovered. Dispose of any unused solvent in organic waste.

Use your TA's comments on the prelab to help your purpose on the post-lab report. For all experiments, print your pre-lab and bring it with you to your section. Your TA will grade it on the spot to determine if you can perform the experiment for the day.

Note: Because there are so many different unknowns that we use in the lab, a table of reagents is NOT NECESSARY for labs 1-5.

Now, go to the schedule of experiments on the course website and find the ChemDraw activity.