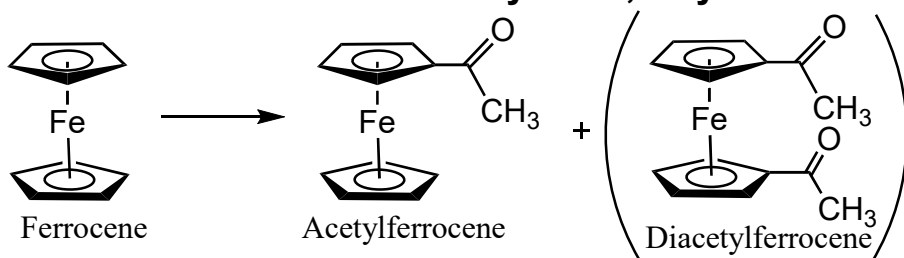


California State Polytechnic University, Pomona

Dr. Laurie S. Starkey, Organic Chemistry Lab II CHM 3150L

Friedel-Crafts Acylation, Day 2



Check-out

- microscale glassware kit
- rubber bulb

Prepare Micropipette

"Spotters" for TLC

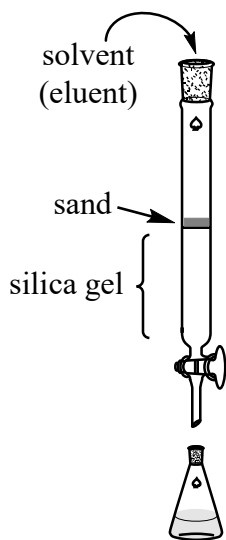
- before starting procedure
- use Bunsen burner to make 4 spotters (use capillary tube or Pasteur pipette)

Procedure: Treat the collected solid with 40 mL of warm hexane to dissolve as much as possible (all of the organic compounds shown above will dissolve, but any polymeric byproduct will not). **Note:** ligroin = petroleum "pet" ether = hexanes (nonpolar mixture of alkanes). **This will be described as the "crude" product solution.** We are going to crystallize our product from this solution, but a small portion of the solution will be removed in order to separate and analyze the reaction mixture, using column chromatography and TLC.

~1-2 mL of
crude solution
(full pipette)
add to column

Crystallization of product

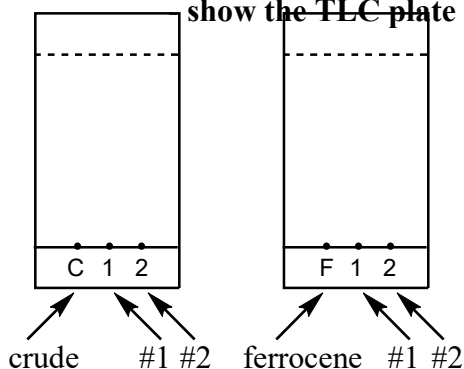
- gravity filter solution into a small beaker to remove insoluble residue (solution should be clear) *note: clear means not cloudy*
- concentrate to ~half volume by boiling off solvent in hood (use a hotplate and add boiling chip or stick to prevent bumping)
- skip decolorizing charcoal step
- cool solution to room temperature and then in ice bath
- collect crystals (vacuum filtration or pipette off liquid)
- **analyze product (yield, mp, IR)**



Separation and analysis of mixture by column chromatography:

- always use rubber bulb to force solvent through quickly: "flash" column
- fill microscale column ~halfway with dry silica gel
- cycle through hexane (use bulb) to "wet" the silica and get out air bubbles
- after packing column, add a 1-2 mL aliquot of the crude mixture to top of silica (use bulb to force solution into the silica - called "loading" the column)
- use enough hexane to elute yellow band (ferrocene - may not be present!) Fraction #1
- use 50% ether/hexane to elute orange band (acetylferrocene) Fraction #2
- note presence or absence of a third colored band remaining on column (diAcferrocene)
- run a TLC plate comparing both fractions with crude solution as reference
- run a TLC plate comparing both fractions with ferrocene solution as reference

show the TLC plate results to instructor and sketch into lab notebook



Compare these two purification techniques.

Recrystallization is quick, easy, and inexpensive, but you sacrifice yield because some product will always stay dissolved in the mother liquor. If we had purified all of the product by column chromatography, it would be more time-consuming and more expensive, but you could isolate each component with nearly 100% recovery, and unlike recrystallization, it is not limited to solid compounds!

TLC eluent (solvent): ~30% ether, 70% hexane (~1:2 ratio)
(~10 mL needed) Record solvent composition in observations