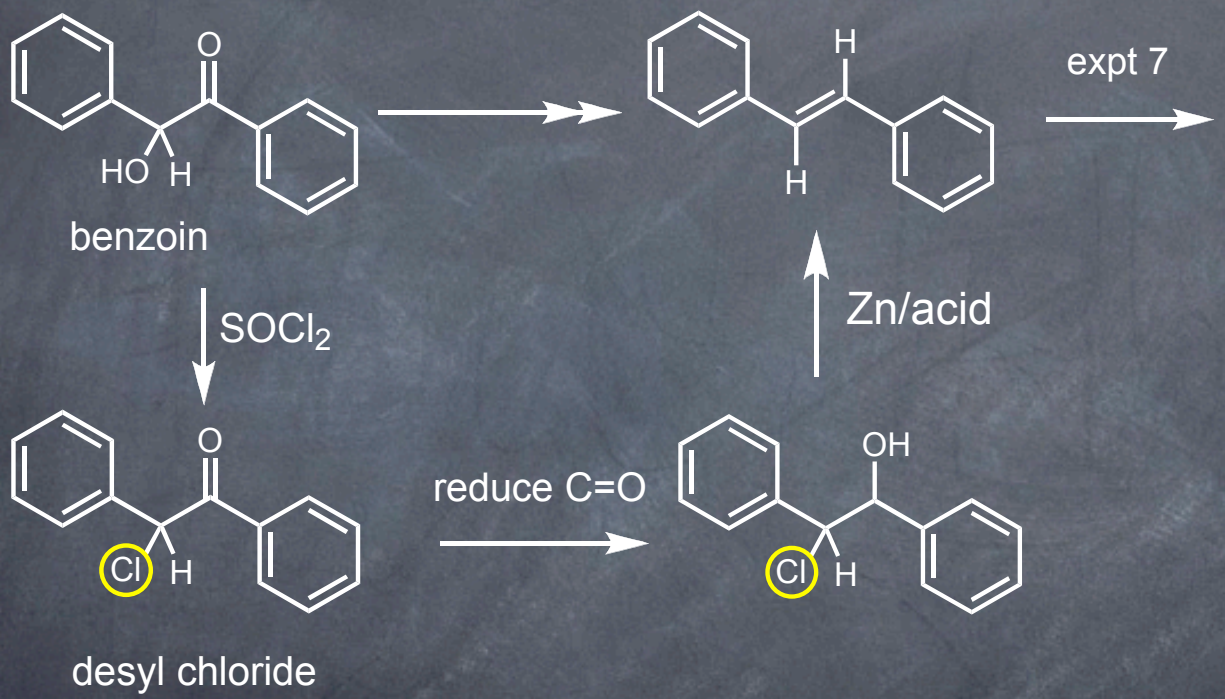
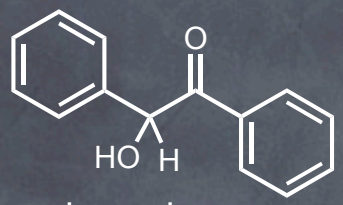
PreLab 1 Experiment 5

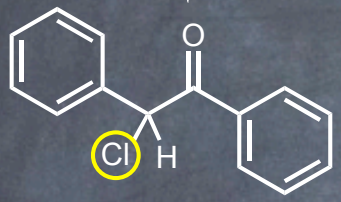


- powerful lachrymator
- ensure ALL solutions/apparatus/gloves stay in hoods
- Take great care also with thionyl chloride



benzoin

$SOCl_2$



desyl chloride

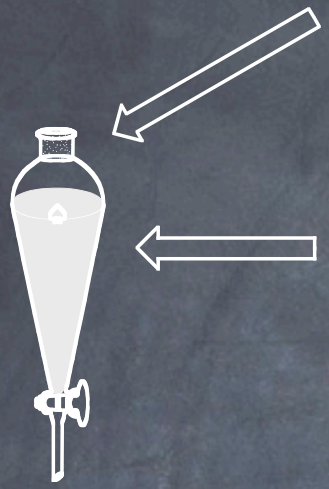
- Heat using a sand bath
- Get this heating FIRST (Saves time!). DO NOT OVEHEAT!!!
- ONLY use SOCl2 in hoods

- **Thionyl chloride**: In Hoods ready with a measuring cylinder. STOPPER your rb flask (contain benzoin) and take this to a hood with thionyl chloride in it.

- Ensure sand is NOT TOO HOT.
  Measure temp before putting flask in!

- Whilst heating (only a few mins) get your other apparatus ready (beaker, sep funnel, 100 mL rb flask..)
- You get an ether solution of desyl chloride (remember: TOP layer during extraction is ether!)
- STOPPER, wear NEW gloves and take to a hood with a rotavap INSIDE it to take off the ether
- STOPPER after evaporation and bring back to your own hood...
- Keep all glassware and solutions in hood and back from front.
- LATER come back and wash glassware ALL in hoods.
- Dispose of gloves and filter paper in bags provided in hoods!

- Keep samples of desyl chloride for IR and UV
- Hint: Make up solution for desyl chloride UV but then get the next stage going!
- Do IR/UV after the reduction if there is a wait for spectrometers



DO NOT!!!!! Clamp the neck!

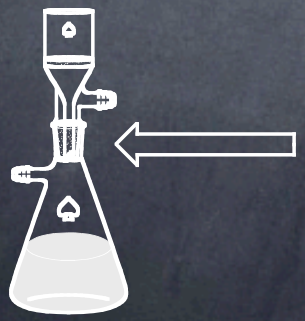
Why? It may slip out or if you clamp too tight it will crack

Support here with an O-ring clamped to a clampstand!

thermometers

check it goes high enough!

Place IN sand sand carefully! Do not use as spear!

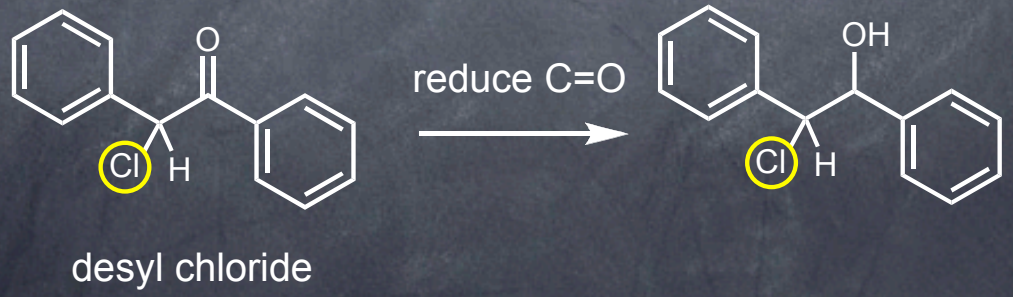


Support here with a clamp clamped to a clampstand!

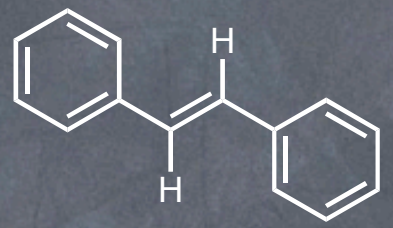
Why? If free standing you may knock it over or it may fall over due to tube tension

- You use sodium borohydride as the reducing agent
- Note: It DOES go off in moist air with time, so do not linger when crushing it, and put the lid on the bottle!

- Hint: You have a 1hr reflux for stage 2 with Zn/acid. Plan use of time.
- FINAL stage is recrystallization from ethanol. Heat this (in a beaker/flask) in a sand bath NOT directly on a hot plate

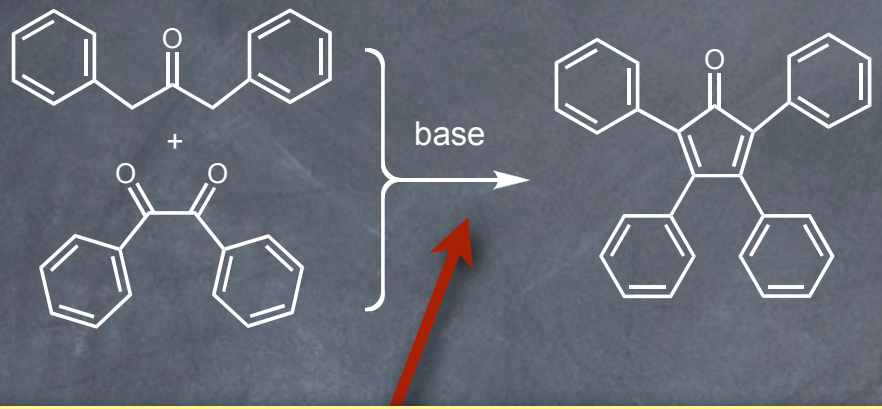


desyl chloride



Zn/acid

PreLab 2 Experiment 6

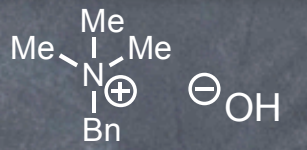


- Dark, very crystalline

- mix reagents, add base then heat in a sand bath
- cool and collect crytalline product! Recrystallze

- You will have time whilst heating, or during the cooling/crystallization, to continue completion of Expt 5, if you have not yet done so already

- If UV machine still dysfunctional, retain sample for analysis next week.



the base

Think during lab:

> What does this do?

> Why is the product so dark/black?